TECHNICAL PROGRESS REVIEW

REACTOR CORE MATERIALS

PREPARED BY **BATTELLE MEMORIAL INSTITUTE** FOR THE U S ATOMIC ENERGY COMMISSION

MARCH 1958 VOLUME 1 NUMBER 1

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Reactor materials.

REACTOR CORE MATERIALS

a review of recent developments in
solid materials for reactor cores

prepared by
BATTELLE MEMORIAL INSTITUTE

MARCH 1958 VOLUME 1 NUMBER 1

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foreword

This is the first issue of a quarterly publication covering significant developments in the field of solid reactor core materials. It is a compilation of reviews of specialized topics prepared by staff members of Battelle Memorial Institute at the request of the Division of Information Services of the U. S. Atomic Energy Commission.

Its purpose is to provide a convenient condensed source of information to two types of readers: (1) those who wish to keep abreast of developments in a general way and (2) those interested in learning where to find detailed accounts of work in particular areas. Complete bibliographies are provided at the end of each section for the benefit of the latter. Coverage has been limited to those core materials which are solid at reactor operating temperatures.

This report differs from a simple compilation of abstracts in several important respects. First, an attempt has been made to concentrate on items that represent recent significant advances or new concepts with good promise. Second, wherever possible, developments in different laboratories along similar lines have been brought together into correlated reviews. Emphasis has been placed on specific accomplishments and their significance, rather than on extensive presentation of detailed results.

R. W. DAYTON
E. M. SIMONS
Battelle Memorial Institute

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Unalloyed Uranium

Current studies of uranium are directed toward knowledge of its physical properties, heat-treatment, and irradiation behavior.

A comprehensive evaluation of data obtained by numerous investigators on the thermal and electric conductivity of uranium is reported by Argonne National Laboratory (ANL).[1,2] Figures 1 and 2 are representative curves of data from many investigators and show thermal conductivity vs temperature and electric conductivity vs temperature, respectively. Investigations are in progress at Battelle Memorial Institute (BMI)[3] to measure the thermal and electric conductivities of uranium before and after irradiation; thus far no data have been obtained.

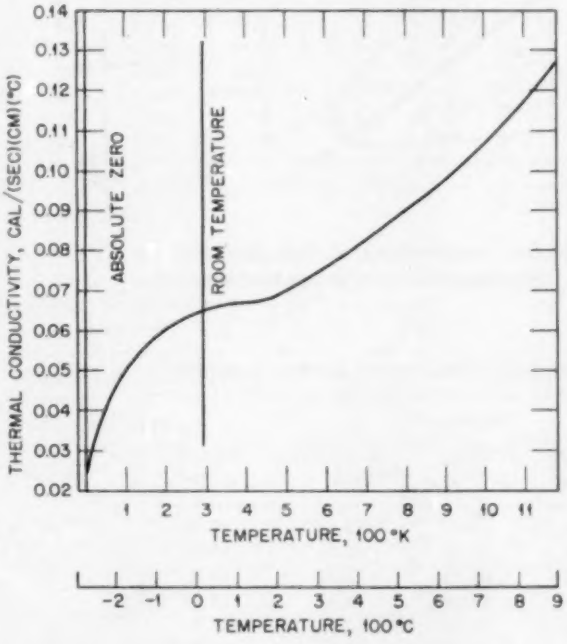

Figure 1—Representative curve of thermal conductivity of ordinary purity beta-treated uranium along rolling direction. Data from various sources compiled by Argonne National Laboratory, Lemont, Ill.[1]

An extensive study of the allotropic transformations in uranium is being conducted at Ames[4] using X-ray, resistivity, and sonic methods. Data from this investigation and others were correlated to give the values reported in Tables I-1 to I-4 for lattice constants, volumes, and densities of alpha, beta, and gamma uranium at different temperatures, as well as transformation characteristics.

True stress-strain curves, for tension and shear of alpha uranium, and yield strength, ultimate tensile strength, Young's modulus, reduction of area, and elongation properties are reported by Bridgeport Brass Co.[5] Formulation of a mathematical model for creep of alpha uranium was carried out by Hanford investigators.[6] The effects of uranium hydride on the tensile properties of uranium are also being studied.[7] The effect of uranium hydride inclusions, either in the grain-boundary network or in massive needle form, although significant, is comparatively small. In general, the presence of uranium hydride causes a 16 to 22°C increase in the brittle-ductile transition temperature. For all tensile testing temperatures (−22 to 200°C), the elongation is lower by 1 to 15 per cent in uranium containing 5 to 6 ppm than in that containing 1 ppm of hydrogen. The ultimate strength of uranium containing 5 to 6 ppm of hydrogen is lower by 3000 to 5000 psi than the strength of uranium with 1 ppm of hydrogen, except for specimens with a grain-boundary network of hydrogen inclusions.

Suggestions applying to beta-transformation treatment of uranium for the attainment of finer grain size are as follows:[8]

1. Introduce a high degree of cold work before beta treatment.
2. Heat rapidly into the beta phase.
3. Heat to a peak temperature well above the alpha-beta transition range.

4. Cool rapidly through the beta-alpha transition range.

5. Hold for the briefest possible time in the temperature range between the beta-alpha transition temperature and the recrystallization temperatures.

The presence of 7.5 ppm of hydrogen in the uranium is conducive of fine grain size. In addition, suggestions for randomization of crystallographic orientation are (1) maintain steep temperature gradients throughout the formative stages of new crystal growths, (2) mechanically work the metal with directional flow while recrystallization is in progress or after it is completed, and (3) externally apply forces causing tensile and compressive stresses on opposite

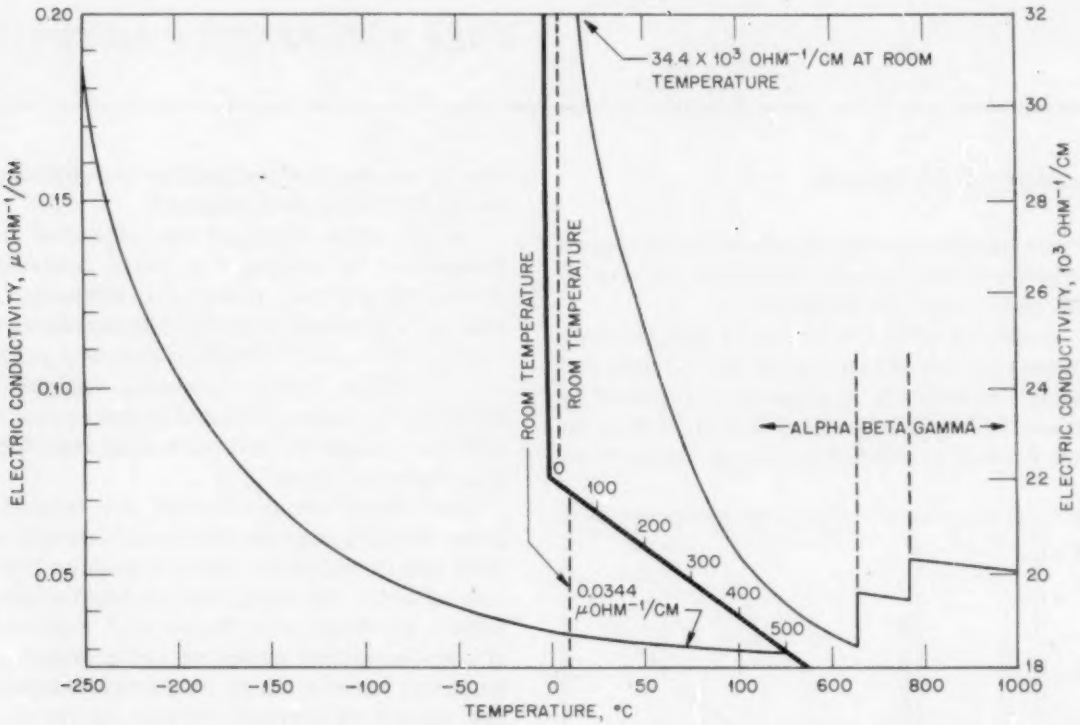

Figure 2—Representative curve showing electric conductivity of beta-annealed unalloyed uranium. Data from various sources are compiled by Argonne National Laboratory, Lemont, Ill.[2]

Table I-1 LATTICE CONSTANTS, VOLUMES, AND DENSITIES OF ALPHA URANIUM*

Temp., °C	Lattice constants			Volume			Density, g/cm³
	a_0	b_0	c_0	Unit cell, 10^{-24} cm³	Atom, 10^{-24} cm³	G-atom, cm³	
0	2.852	5.864	4.952	82.84	20.71	12.47	19.08
25	2.853	5.864	4.954	82.91	20.72	12.48	19.07
100	2.858	5.864	4.961	83.16	20.79	12.52	19.01
200	2.866	5.864	4.972	83.56	20.89	12.58	18.92
300	2.874	5.862	4.985	84.01	21.00	12.65	18.82
400	2.885	5.859	5.000	84.53	21.13	12.72	18.70
500	2.896	5.851	5.018	85.07	21.26	12.80	18.58
600	2.910	5.839	5.040	85.64	21.41	12.90	18.45
662	2.920	5.831	5.054	86.07	21.51	12.66	18.36

*Data compiled by Ames Lab., Iowa State College, Ames, Iowa.[1]

Table I-2 LATTICE CONSTANTS, VOLUMES, AND DENSITIES OF BETA URANIUM*

Temp., °C	Lattice constants a_0	Lattice constants b_0	Volume: Unit cell, 10^{-24} cm^3	Volume: Atom, 10^{-24} cm^3	Volume: G-atom, cm^3	Density, g/cm^3
662	10.744	5.651	652.4	21.74	13.09	18.17
675	10.748	5.651	653.2	21.77	13.11	18.15
700	10.754	5.652	654.0	21.80	13.13	18.13
725	10.760	5.653	654.8	21.82	13.14	18.11
750	10.761	5.654	655.4	21.84	13.15	18.09
772	10.771	5.655	656.1	21.87	13.17	18.07

*Data compiled by Ames Lab., Iowa State College, Ames, Iowa.[4]

Table I-3 LATTICE CONSTANTS, VOLUMES, AND DENSITIES OF GAMMA URANIUM*

Temp., °C	Lattice constant, a_0	Volume: Unit cell, 10^{-24} cm^3	Volume: Atom, 10^{-24} cm^3	Volume: G-atom, cm^3	Density, g/cm^3
772	3.532	44.06	22.03	13.27	17.94
800	3.534	44.13	22.06	13.29	17.91
850	3.538	44.28	22.14	13.33	17.85
900	3.542	44.43	22.21	13.35	17.79
950	3.545	44.58	22.29	13.42	17.73
1000	3.549	44.73	22.36	13.47	17.67
1050	3.553	44.87	22.43	13.51	17.61
1100	3.557	45.01	22.50	13.55	17.56

*Data compiled by Ames Lab., Iowa State College, Ames, Iowa.[4]

Table I-4 TRANSFORMATION CHARACTERISTICS*

Alpha-Beta			Beta-Gamma		
Equilibrium Temp., 662°C			Equilibrium Temp., 772°C		
Cooling:			Cooling:		
α_s = 658 ± 5°C†			β_s = 768 ± 4°C		
α_f = 645 ± 5°C‡			β_f = 761 ± 3°C		
Heating:			Heating:		
β_s = 666 ± 2°C			γ_s = 776 ± 3°C		
β_f = 690 ± 10°C			γ_f = 786 ± 4°C		
Temp., °C	Per cent transformed	Time, min	Temp., °C	Per cent transformed	Time, min
666	0		776	0	
667	25	46.5	777	53	14.0
668	38	17.0	778	73	11.5
670	55	8.8	779	81	10.0
678	87	2.0	780	86	9.0
684	96		784	95	
690	100		786	100	

* Data compiled by Ames Lab., Iowa State College, Ames, Iowa.[4]

† Temperature above which no isothermal transformation is detectable.

‡ Temperature above which no isothermal transformation is complete (conversely on heating).

sides of the uranium rod or slug during the time that crystal growth is occurring.

Uniform heating and cooling is essential in heat-treatment to prevent asymmetric stresses from being set up, with accompanying asymmetry of orientation zones. It should be emphasized that the conditions most conducive to fine grain size tend to promote preferred orientation so that a compromise between the two desiderata may be the best that can be done.

Data on the effect of irradiation and postirradiation annealing on the room-temperature tensile properties of uranium[9,10] are presented in Table I-5. For uranium irradiated at 150°C at exposures from 0.03 to 0.1 at. % burn-ups, the damage is essentially saturated at the lower exposure level. Postirradiation alpha-phase annealing can result in substantial recovery in ductility. The amount of the recovery obtainable decreases with increasing exposure rates. Postirradiation gamma-phase annealing with slow heating and cooling throughout the transformations resulted in material with appreciable ductility. (M. S. Farkas)

Alpha-Uranium Alloys

Recent developments in the field of alpha-uranium alloys include constitutional-diagram studies and corrosion, radiation effects, and dimensional-stability tests.

Ames reports the following results in their study of uranium-rich alloys of the uranium-antimony system: the alpha-to-beta transformation is raised to 674°C, and the beta-to-gamma transformation takes place isothermally at 781°C. There is apparently a eutectic near 0.1 wt. % antimony and a high-temperature eutectic[11] between U_5Sb_3 and USb.

Further investigation of the high-uranium portion of the uranium-titanium phase diagram is being performed at BMI.[12]

The determination of the UPb structure was carried out, and an explanation of methods employed in determining the extent of liquid immiscibility in the uranium-lead system has been published.[13]

Corrosion studies[14] of uranium-titanium alloys at ANL show that apparently a minimum of 2.7 wt. % titanium is required for reasonable corrosion rates at 290°C. The corrosion behavior of the gamma-quenched alloy at 290 and

Table I-5 EFFECT OF IRRADIATION AND POSTIRRADIATION ANNEALING ON THE ROOM-TEMPERATURE TENSILE PROPERTIES OF URANIUM*

Exposure burn-up, at. %	Annealing temp., °C	Annealing time, hr	Ultimate tensile strength, 1000 psi	Yield strength, 0.2 % offset, 1000 psi	Elongation in 1 in., %
Control			97.1	37.8	19 ± 1
0.018	†		78.0 av.	57.0 av.	0.75 av.
0.018	400	10	75	39.2	2.5
0.018	400	25	76	39.2	2.6
0.018	400	100	74.9	48.1	1.6
0.018	600	10	84.9	35.2	5.7
0.031	†		83 av.	73 av.	0.7 av.
0.031	400	100	79.2	56.6	1.1
0.031	600	10	82.3	42.2	2.7
0.075	†		88.0 av.	79.0 av.	0.5 av.
0.075	400	10	80.7	62.8	0.7
0.075	400	25	77.7	57.2	0.7
0.075	400	100	82.3	63.5	0.8
0.075	600	10	87.5	53.0	1.2
0.075	600	25	81.0	70.5	0.9
0.075	600	100	87.2	50.6	1.2
0.075	700	10	52.5 av.	40.8 av.	0.95 av.
0.075	700	3‡	40.0	24.3	1.14
0.075	700	10§	49.1	18.7	2.1
0.075	800	10§	36.1	17.3	12.5
0.10	†		77.4 av.	73.4 av.	0.55 av.
0.10	800	10§	32.2	29.1	4.6

* Data from Hanford Atomic Products Operation, Richland, Wash.[9,10]
† As irradiated.
‡ Heated at 10°C/hr – held 3 hr, furnace cooled (approximately 150°C/hr).
§ Heated and cooled from 600°C at 10°C/hr.

315°C is summarized in Table I-6. There is evidence that the material was not homogeneous.

Studies[15] on a uranium–6 wt. % zirconium alloy in 60 and 90°C water under rapid-flow conditions show corrosion rates of 0.9 and 0.7 mg/(dm^2)(day), respectively, at periods of exposure up to 60 days. There appears to be little effect of the flow rate of water.

Table I-6 CORROSION OF GAMMA-QUENCHED URANIUM–2.7 WT. % TITANIUM ALLOY*

Temp., °C	Time, days	Corrosion rate, mg/(cm^2)(day)	Remarks
290	5	24	Uniform corrosion
	5.8	31.8	Uniform corrosion
	5.9	39.0	Sample appears to be dividing into layers
	6.9	54.4	Sample appears to be dividing into layers
315	3.8	77.2	Pitting
	6.8	116.2	Test stopped

* Data from Argonne National Laboratory, Lemont, Ill.[14]

Nuclear Metals, Inc.,[16] is investigating a diffusion-anneal technique employing uranium–2 wt. % zirconium clad with Zircaloy-2 for better corrosion life. To test the efficiency of the diffusion-produced layer, a 0.005-in.-diameter hole was drilled through the clad and just barely to the core (on the cylindrical surface). Specimens so defected (both before and after heat-treatment) showed no change after testing in water for 21.8 days at 290°C, followed by 31.7 days at 350°C. Samples given a similar treatment showed no metallographic evidence of core corrosion. Samples defected with 0.016-in.-diameter holes showed widely varying corrosion lifetimes. A sample irradiated to a burn-up of approximately 0.1 at. % (defected before heat-treatment) showed no apparent changes after corrosion testing for 2.6 days at 290°C and then for 9.2 days between 315 and 338°C.

The diffusion heat-treatment definitely produced a protective barrier on the uranium–2 wt. % zirconium alloy under the conditions of

test. This protection is apparently unaffected by relatively small amounts of nuclear radiation.[14,17]

Table I-7 GROWTH OF URANIUM ALLOYS UNDER IRRADIATION AT ELEVATED TEMPERATURES*

Specimen comp., wt. %	Estimated burn-up, at. %	Temp., °F		Diameter increase, %	
		Max.	Normal	Av.	Max.
U–2 Zr	0.25	640	600	4	4
U–1.2 Mo	0.3	790	790	9	10
U–1.2 Mo	0.4	1050	570	12	13

*Data from Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.[18]

Irradiation tests conducted by Atomics International Div.[18] are reported in Table I-7. The growth recorded is indicative of the problem existing in the elevated-temperature irradiation of solid uranium fuels containing small amounts of alloying elements.

Table I-8 THERMAL-CYCLING DATA FOR URANIUM ALLOYS*

Alloy additions, wt. %	Density, g/cm³			Changes in 500 cycles, %	
	Theoret.	Before cycling	After cycling	Diam.	Height
0.5 Nb	18.88	18.21	11.97	+19.6	+25.4
1.6 Nb	18.64	17.71	15.79	+8.0	+3.6
6 Nb†	17.71	17.45	17.43	+0.8	+0.3
10 Nb	16.84	15.62	15.45	+0.3	+0.3
0.5 Nb + 0.5 Al	18.33	17.41	11.30	+28.9	+9.3
1 Nb + 0.25 Si	18.45	17.39	10.64	+32.4	+41.0
2 Mo + 0.5 Zr	18.5	17.66	17.92	+0.2	−0.7

*Data from Sylvania Electric Products, Inc., Bayside, N. Y.[20]

†Arc-melted alloy.

Investigations at Hanford[19] showed that alloying uranium with 1.5 at. % silicon increases the ultimate tensile and yield strengths by 50 per cent with a loss in ductility of about 25 per cent. Several extruded alloy fuel elements of this composition were irradiated to 1007 Mwd/ton to determine if increasing the strength of the uranium increases its ability to withstand irradiation. Examination of the alloy fuel elements after irradiation revealed a greater tendency to warp and to fracture transversely than unalloyed uranium.

Dimensional-stability results[20] for uranium alloy powder compacts, on alpha-beta cycling, are partially reported in Table I-8. The stability of 2 wt. % molybdenum–0.5 wt. % zirconium appears very good. Aluminum or silicon is ineffective as an addition element in conjunction with low niobium contents. (M. S. Farkas)

Gamma-phase Uranium Alloys

Uranium-Niobium Alloys

The removal of carbon through zirconium additions and subsequent zirconium carbide flotation has been attempted at Nuclear Metals.[16] Studies at BMI indicate carbon gettering by Zircaloy cladding.

The transformation kinetics of uranium-niobium base ternary alloys have been studied at Armour Research Foundation.[21,23] Times for initial hardness change which are indicative of transformation in the alloys are found to increase with chromium, vanadium, and ruthenium additions and to decrease with titanium, nickel, and silicon additions. Studies of the martensite transformation at Bettis indicate that, at about 6 wt. % niobium, the M_S is depressed below room temperature.

Uranium-Molybdenum Alloys

Creep-rupture data for uranium–6 to 12 wt. % molybdenum alloys at 1300°F are reported by BMI. The minimum creep rate under 500 psi stress decreases from 0.0024 to 0.0016 per cent/hr as the molybdenum content is increased from 8 to 12 wt. %. The creep rate for the 12 wt. % alloy showed no change due to decreased stress level from 500 to 200 psi. At 1500°F and higher, the alloys creep under their own weight, and intergranular separation occurs. Linear thermal-expansion data to 950°C for these same alloys are also reported, as is the thermal conductivity to 900°C of the uranium–8 wt. % molybdenum alloy. Mean linear-thermal-expansion coefficients between 20 and 900°C of approximately 17.2×10^{-6}, 16.1×10^{-6}, 15.8×10^{-6}, and 16.4×10^{-6}/°C for the 6, 8, 10, and 12 wt. % molybdenum alloys, respectively, were recorded. The thermal conductivity of the uranium–8 wt. % molybdenum alloy increased from 0.111 at 20°C to 0.420 watt-cm/(cm²)(°C) at 900°C.

Lattice-parameter measurements for the retained gamma phase in uranium-molybdenum alloys are reported by ANL[21] to extrapolate to a lattice constant of 3.495 A for pure gamma uranium. This differs from the 3.474 A determined in a previous study at Bettis Plant.

(A. A. Bauer)

Epsilon-phase Uranium Alloys

Uranium-Zirconium Alloys

Studies of structure, diffusion, transformation kinetics, and stress-strain curve anomalies have been reported by Knolls Atomic Power Laboratory (KAPL), Bettis, and BMI. KAPL[10] reports the structure of epsilon to be primitive hexagonal, based on the composition Zr_2U, with the zirconium and uranium atoms assuming ordered positions in the lattice. Kinetic data for epsilon formation have been obtained at Bettis[22] and BMI;[23] a diffusion-controlled nucleation and growth process is postulated for the transformation from the gamma-uranium solid solution. A C-curve with the nose lying between 400 and 500°C, constructed[22] on the basis of dilation data, shows the transformation to proceed extremely rapidly. Approximately 2 sec at the nose is required for 25 per cent transformation, whereas the transformation is completed within 100 min. Diffusion coefficients for uranium-zirconium diffusion at 1000°C are reported by Bettis,[21] as calculated by the Matano method. The values determined are given in the following tabulation and are in good agreement with those reported earlier by Adda and Philibert (French workers).

Uranium, at. %	Diffusion, 10^{-10} cm²/sec
5	9.2
10	6.2
20	5.7
25	5.1
30	5.7
40	6.6
50	7.5
60	10.3
75	16.1
85	25.2
90	49.8

The reaction of the uranium–50 wt. % zirconium alloy with oxygen between 200 and 500°C has been studied at Bettis.[24] Oxidation, in general, followed a linear rate law. At 400°C and above, transformed epsilon-phase specimens oxidized more rapidly than did gamma-quenched and gamma-rolled specimens. Since gamma transforms to epsilon rapidly at these temperatures, this effect cannot be related to difference in structure.

Corrosion studies of Zircaloy-2 clad uranium–50 wt. % zirconium specimens in 680°F water have been reported by BMI, and heat-treatments designed to improve the corrosion resistance of the core-to-clad bond are described.[12] Water quenching from 800°C appears to destroy corrosion resistance. Artificially defected Zircaloy-2 clad uranium–50 wt. % zirconium plates are found to corrode evenly, with no evidence of discontinuous failure when epsilonized. Specimens of this type have been on test for 588 days in 650°F water with only a small blister, approximately ¼ in. in diameter, appearing around the defect. The corrosion resistance of the diffusion zone between core and clad appears to be equal to or better than that of the core itself.[73]

Uranium-Silicon Alloys

Corrosion tests performed on unirradiated and irradiated samples of uranium–3.8 wt. % silicon in 500 to 600°F water are reported by ANL.[14,23] Corrosion behavior prior to irradiation was found to be variable. Apparently, lack of homogeneity presents a problem in this alloy. Irradiated samples were found to exhibit short life in corrosion, the samples tending to crack and split. Stresses introduced as a result of irradiation may be responsible for this behavior. Another possible explanation earlier proposed by Bettis is de-epsilonization as a result of radiation.

(A. A. Bauer)

Dilute Uranium Alloys

Aluminum-Uranium Alloys

Effort is being devoted to the casting of sound, homogeneous aluminum-uranium alloys containing up to 50 wt. % uranium. The major difficulties encountered in the casting of the high alloys are porosity and inhomogeneity. Battelle[25] has reported the production of sound, homogeneous 45 wt. % uranium alloy extrusion billets by vacuum induction melting and gravity casting, and work

is continuing. In addition, the production of high-alloy extrusion-billet castings by centrifugal casting in air[3,25] is being studied.

Oak Ridge[10] reports the effects achieved by the addition of 3 wt. % silicon to a 48 wt. % uranium alloy. The silicon seems to suppress the formation of UAl_4 and allows the primary nucleating phase [$U(Al,Si)_3$] to be retained as the stable phase. This reduces the volume fraction of compound present, refines the grain size, reduces hot tearing and shrinkage, and reduces edge cracking. Ternary additions of 3 wt. % germanium, zirconium, and strontium also suppress UAl_4 formation. (It would seem that additions other than silicon might be more desirable because silicon causes difficulties in chemical reprocessing.) Work at BMI[25] shows that silicon in amounts up to 0.5 wt.% has no effect on the melting characteristics of the 16 wt. % uranium alloy. Work at Nuclear Metals[27] on extrusion of aluminum-uranium alloy tubes shows that large compound size along with nonuniformity result in poor ductility.

Table I-9 METALLOGRAPHIC ETCHING TECHNIQUES

Etchant	Application	Identification
	Al-UAl_4 field	
1 part 40% chromic acid + 1 part 50% acetic acid	Electrolytic, 3 min at 0.05 amp/cm^2	UAl_3, yellow UAl_4, blue-gray
50% HNO_3 + H_2O	Chemical, 1-min immersion	UAl_3, bright show UAl_4, gray
	UAl_3 + UAl_4 field	
The above etchants when used on material of this composition are satisfactory. The chromic electroetch colors UAl_3 orange and the nitric etchant colors the UAl_4 light.		
	UAl_2 + UAl_3 field	
The chromic electroetch darkens and pits UAl_2 and colors the UAl_3 bright yellow. The nitric etchant colors UAl_2 light blue and colors UAl_3 light yellow. A chemical etchant of 1 wt. % HF in water darkens UAl_3.		

Studies in progress at BMI[26] indicate that magnesium has a coarsening effect on the UAl_4 particles, whereas silicon in amounts up to 1 wt. % produces finer particles. There is very little evidence that silicon at the 1 wt. % level suppresses the formation of UAl_4. It is reported[3] that increasing magnesium contents (up to a total of 0.5 wt. %) increases the room-temperature hardness of the alloys. Harwell reports that up to a uranium content of ~68 wt. %, the room-temperature equilibrium structure[28] is Al + UAl_4. Alloys containing between about 68 and 74 wt. % uranium should be UAl_3 + UAl_4 with no free aluminum. The formation of UAl_4 was suppressed by a fast cooling rate through the critical range. During the course of this study, metallographic etching techniques (Table I-9) were developed for the identification of the various uranium-aluminum compounds.

Battelle[25] reports the corrosion resistance of alloys containing up to 45 wt. % uranium to be comparable to that of the 16 wt. % uranium alloy in 300°F water. Metallographic studies indicated no diffusion of alloys containing up to 45 wt. % uranium into 2S (1100) aluminum after 30 days at 750°F.

Palladium-Uranium Alloys

The National Bureau of Standards (NBS) reports[10,21] that the solid solubility of uranium in palladium is on the order of 30 wt. % and that certain anomalies are present in the 30 to 36 wt. % palladium range. Alloys containing nominally 30 to 32 wt. % palladium show a strong arrest at 950°C, whereas an alloy containing 36 wt. % palladium (nominal) shows a sluggish arrest at 950°C. These data are being rechecked; the alloy composition could be uncertain.

Ruthenium-Uranium Alloys

Argonne reports investigation[21] of the ruthenium-uranium system in the range 20 to 50 wt. % ruthenium. Also reported is the probable existence of six compounds across the diagram U_2Ru, URu, U_3Ru_4, U_2Ru_3, U_3Ru_5, and URu_3. Diffraction patterns have been obtained for U_2Ru, URu, and U_3Ru_4.

The melting of alloys between 57 and 94 wt. % ruthenium at about 1850°C is reported by NBS.[10,21] Further work indicates the possibility of a eutectic reaction at this temperature. The possibility of a high melting compound, URu_3 or URu_4, is also mentioned.

Zirconium-Uranium Alloys

Bettis[29] concludes the following about the corrosion behavior of low uranium-zirconium alloys in static 680°F water and 1500 psi, 750°F steam.

1. Corrosion of alloys containing 4 to 15 wt. % uranium is characterized by formation of a

protective oxide film, followed by a breakdown and general spalling of the film.

2. Corrosion resistance decreases with increase in uranium.

3. Mechanism of corrosion may be different in high-temperature water and in steam.

4. Substitution of Zircaloy-2 for sponge zirconium in the alloy does not improve corrosion resistance; however, the addition of 0.5 to 2.5 wt. % tin slightly improves resistance of the alloy.

Evaluation of 6.4 wt. % uranium alloys is in process at BMI. Metallographic examination of irradiated specimens revealed no visible effects of radiation on the bond nor on the core. The cladding exhibited normal distribution of hydrides. The average results of bend test performed on the irradiated material are

Test temp. (room):
Modulus of elasticity, 11.4×10^6 psi
Yield strength (0.2% offset), 39,000 psi
Test temp. (600°F):
Modulus of elasticity, 7.1×10^6 psi
Yield strength (0.2% offset), 32,700 psi
Ultimate tensile strength, 45,100 psi
Strain at fracture, 6.2%

Knolls[30] reports results of irradiation-damage studies performed on twisted oval ribbons of 7, 12, and 22 wt. % uranium alloys clad with Zircaloy-3. An assembly of eighteen 22 wt. % uranium–zirconium alloy elements received a burn-up of 1.9 at. % with only one element exhibiting a failure—this was a lenticular type fracture about ½ in. long on the high-flux end. Fifteen 7 wt. % uranium alloy elements were given a burn-up of 1.3 at. % without failure. Single pins of 8 and 7 wt. % uranium alloy have been given burn-ups of 1.8 and 1.5 at. %, respectively (analyzed), at core temperatures between 890 to 900°F without failure. On the other hand, two specimens of 7 wt. % uranium alloy[31] have failed after burn-ups of 1.5 and 1.9 at. % at calculated core temperatures of about 1100°F. This failure was cracks in the cladding, which appeared to start at the surface of cladding and worked inward. The cracks were transgranular and did not appear to start from any particular defect. On the basis of data presented, it would seem that temperature of irradiation has more effect than burn-up.

Eighteen 22 wt. % uranium alloy ribbon elements were irradiated to a burn-up of 1.9 at. % in zone of maximum flux (analyzed). No core temperature was reported; however, it was reported that the test was equivalent to full power endurance of 3100 to 3200 hr. The mean increase in the minor axis of the specimens was 5.4 mils; the major axis decreased by a mean of 0.7 mil. The oval shape provides conditions for the build-up of unequal stresses, which may account in part for the cracks in the clad mentioned above.

Battelle[32] reports that the phase boundary of the alpha-plus-beta zirconium region occurs at 31 wt. % uranium at 660°C and also proposes revisions to the zirconium-uranium-oxygen and the zirconium-uranium-nitrogen diagrams. In another study[21] a 22 wt. % uranium–zirconium alloy, treated to produce a structure having alpha zirconium in grain boundaries surrounding dispersed epsilon, appeared more resistant to radiation-induced warpage than did other structures of the 22 wt. % uranium alloy. These were irradiated at about 400°C to burn-ups of about 0.95 at. %. Alloys containing 50 wt. % uranium[3] are being irradiated in two conditions of heat-treatment at a temperature of 650°C in the hope of obtaining a burn-up of 2 at. %. In one condition (water quenched from 850°C), the grain size is 0.021 mm, and, in the other (cold swaged, heated 24 hr at 500°C, furnace cooled, heated to 625°C, and water quenched after 10 min), the grain size is 0.003 to 0.004 mm. It is hoped to determine the effect of grain size on radiation stability.

Niobium-Uranium Alloys

Battelle prepared alloys of niobium containing 20 and 25 wt. % uranium with little difficulty. Some difficulty was experienced in fabricating by hot rolling at 2000°F. Reductions of 45 per cent caused slight edge cracking. It is believed that purity of starting stock is important for fabricating. These alloys were exposed to liquid NaK at 1500°F for eight days without indication of corrosion.

Molybdenum-Uranium Alloys

Battelle has performed limited work with alloys of molybdenum and 20 or 25 wt. % uranium. These alloys can be arc-cast by carefully controlling the cooling temperature, but no satisfactory fabrication procedure was developed during the very brief study. (R. F. Dickerson)

Plutonium Alloys

Study of plutonium is in progress at several laboratories in this country and in England, Canada, and Russia. The most significant current work consists largely of various alloy studies performed at Argonne and Harwell. Much of the research at these laboratories appears to be a part of the respective programs for the EBR-II and the Dounreay fast breeder reactors, and it is apparent from the information available that the programs at the two laboratories are proceeding along somewhat similar technical lines. Some additional information is also available.

Uranium-Plutonium-Molybdenum and Uranium-Plutonium-Fissium Alloys

At ANL[10,14,33] research is in progress on both uranium-plutonium-molybdenum and uranium-plutonium-fissium type alloys. These studies are an outgrowth of earlier work on uranium–20 wt. % plutonium alloys. This alloy was found to have very poor metallurgical properties owing to the presence of small amounts of the brittle zeta phase. The addition of molybdenum to uranium suppresses the formation of the zeta phase and stabilizes the gamma phase which is isotropic and has good metallurgical properties.

The uranium-plutonium-fissium alloys are made up to represent pyrometallurgically reprocessed fuel. In the pyrometallurgical reprocessing, certain of the fission-product elements, including molybdenum, palladium, rhodium, ruthenium, technetium, and zirconium, cannot be removed and tend to build up to an equilibrium amount after several successive recycles of the fuel. The major elements of this group were included in the fissium type alloys for study, and additions totaling 5.4 and 10.8 wt. % of these elements were made to the uranium–20 wt. % plutonium base alloy in ratios corresponding to the pyrometallurgical reprocessing operation. The specific additions totaling 5.4 wt. % were 1.40 wt. % molybdenum, 0.25 wt. % zirconium, 2.15 wt. % ruthenium, 0.35 wt. % rhodium, and 1.25 wt. % palladium. The alloy containing 10.8 wt. % fissium had twice these amounts of the respective elements. In addition, a similar alloy with 5.4 wt. % fissium with extra molybdenum was studied. Fissium also tends to stabilize the gamma phase.

Both the molybdenum- and fissium-containing alloys appear promising and have fairly good metallurgical properties. However, these alloys show a type of instability that manifests itself in the production of varied thermal-expansion coefficients during repeated thermal cycling. The alloys do not change dimensionally, however, during thermal cycling 200 times between room temperature and 500°C. Various data available include thermal-expansion coefficients, density, and hardness.[14]

Data are also reported by ANL on the results of irradiation of the uranium–20 wt. % plutonium–5 wt. % molybdenum and 5.4 and 10.8 wt. % fissium alloys.[14,17] All the specimens show good dimensional stability with exceptionally good surface finish. It was observed that molybdenum without the other fissium elements seems to have slightly less embrittling effect than fissium, although the binary uranium-plutonium alloy is much more brittle than with either of these additions. Softening was observed consistently in the uranium–20 wt. % plutonium–5 wt. % molybdenum alloy as a result of irradiation. The reason does not appear to be too clear. These irradiations were performed at burn-ups of less than 1 per cent of all atoms. There was some scatter in the volume changes, but the changes were consistently low and ranged on either side of what might be expected theoretically.

Thorium-Plutonium Alloys

Considerable data are reported by ANL on thorium–5 to 40 wt. % plutonium alloys.[10,14] Included are details of castability, fabricability, dilatometric, hardness, density, and thermal-cycling studies. The maximum solubility of plutonium in thorium is about 35 at. %, decreasing with decreasing temperature. No major metallurgical difficulties are indicated, but, as in the other alloys, an instability is evidenced by varying thermal-expansion coefficients as the specimens are thermally cycled up to six times. Generally, the thermal-expansion coefficients decreased with increased number of cycles for the lower alloys, but erratic behavior was observed in higher alloys containing 20 to 25 wt. % plutonium. These data indicate that the as-cast condition is not the optimum condition for stability, even though no growth was observed. Alloys containing 5 and 10 wt. % plutonium were also cycled 200 times between room temperature and 500°C, with no dimensional

changes being observed. Additional details of the constitution of thorium-base plutonium alloys are reported.[14]

Additional Research

A recent report[34] describes the plutonium research facilities available at Harwell.

A report from Russia[35] indicates that thin films of plutonium have been produced by electrodeposition from tetrachloride solutions. Satisfactory films up to 0.3 mg/cm^2 were obtained.

(F. A. Rough)

Thorium Alloys

Constitutional-diagram studies at BMI[36] and at ANL[14] show that approximately 1 wt. % uranium is soluble in thorium at 800 and 900°C and that 22 at. % plutonium is soluble in thorium at 400°C. Hardness increases were noted with the addition of either uranium or plutonium to thorium.

Uranium (5 wt. %) increased the hardness of thorium from 45 to 82 DPH. Further additions of uranium up to 20 wt. % produced no additional hardness increase. A 5 wt. % plutonium alloy had a hardness of 75 Rockwell F, whereas a 20.4 wt. % plutonium alloy had a hardness of 94 Rockwell F.

The work at ANL[10] on thorium-plutonium alloys has been discussed in the preceding section on plutonium. The irradiation behavior of thorium alloys containing up to 5.5 wt. % uranium was studied.[37] It was found that, at an irradiation temperature of 100 to 200°C, minor dimensional and hardness changes occurred after burn-ups ranging from 2 to 4.4 at. %. One 1.4 wt. % uranium alloy was still ductile after 2.4 at. % burn-up. More recently, Atomics International[10] found that thorium–10 wt. % uranium alloys were dimensionally stable in irradiations (burn-ups not yet available), producing central temperatures as high as 650°C, and it is planned to extend the experiments[18] to 750°C.

Hanford plans to study the effect of irradiation on mechanical and physical properties of thorium-uranium alloys containing 1, 4, and 5.4 wt. % of highly enriched uranium.[10] The 5.4 wt. % uranium alloy was slightly cold-worked and swaged to a reduction of about 60 per cent, and its recrystallization was studied. It was found that, after 2 hr at 700°C, recrystallization was apparent around oxide stringers, and that, after 2 hr at 800°C, recrystallization was complete except in the center of 0.515-in.-diameter rod. Complete recrystallization occurred after 2 hr at 900°C.

Difficulties have been encountered in finding a suitable cladding material for thorium and its alloys, and apparently zirconium is the only satisfactory answer. Oak Ridge[38] reports that in pressure bonding tests there is no bonding of thorium and aluminum at temperatures below 350°C. Bonding occurs at higher temperatures, but, at a bonding temperature of 600°C, a hard, brittle layer was observed at the bond interface. On the other hand, Nuclear Metals,[39] without stating the process used, reported that good bonding was achieved between Zircaloy-2 and thorium. Chisel tests on Zircaloy clad thorium produced failure in the thorium rather than in the bond or in the cladding. Oak Ridge[38] reports that 750 to 850°C is a suitable temperature range for pressure bonding zirconium and thorium.

(W. Chubb)

Ceramic Fuel and Fertile Materials

Uranium Dioxide Fabrication

Uranium dioxide is one of the most important ceramic fuels.[40,41] Recent reviews cover both the basic properties and technology[42,43] of UO_2 and its performance as a reactor fuel.[44–47]

Dust pressing and extrusion both have been shown to be flexible processes for forming UO_2 ceramic shapes;[44,48–51] the choice of method would depend largely on the size and shape of the pieces required and on the batch size.

The development work being done currently is directed primarily toward improving the sintering characteristics of UO_2 powders. There seems to be good evidence that the ease with which high density can be obtained depends on the method used to prepare the oxide powder.[48,52]

For powder prepared by a given process, workers at Hanford[48] showed that sintered density increases with decreased particle size and increased surface area of the powder, as is the case with other ceramics.

The powders investigated varied in average particle diameter from 0.55 to 4.80 μ and in surface area from 0.42 to 5.19 m^2/g. Densities as high as 95 per cent of theoretical, for powders pressed at 30 Tsi and sintered in hydrogen at

1750°C for 8 hr, were reported. Similarly, Bettis[44,54] reported that ball milling improved the sintered density and the uniformity of the sintered pieces. The sintered density of the pellets made from the ball-milled powder was found to be independent of the pressed density (if the pressed density was above 50 per cent of theoretical). It was also reported[49] that the addition of less than 0.5 per cent of TiO_2 improved sintered density. CeO_2 also has been shown to promote densification in UO_2 ceramics.[55]

An entirely different approach to UO_2 fuel-element fabrication consists in loading dense UO_2 particles into a metal tube and then swaging the assembly. Quinlan and Roake[53] have fabricated elements by this technique, in which the UO_2 was compacted to a density higher than 85 per cent of theoretical by cold swaging 0.675-in.-O.D. stainless-steel pipe to a finished diameter of 0.367 in. O.D. This method appears to hold promise of greatly reduced fabrication costs, provided the thermal and mechanical properties of elements formed in this manner are found to be satisfactory. (H. D. Sheets)

Physical and Chemical Properties of Uranium Dioxide

The physical and chemical properties of UO_2 are being investigated by several laboratories. Many UO_2 properties are sensitive to preparation history and impurities. High-temperature properties are particularly difficult to measure because UO_2 bodies exhibit poor thermal-shock behavior. Battelle has studied the effects of additives (>5 vol. %) on the mechanical and corrosion properties of sintered UO_2 bodies.[52] Of all additives studied, BeO produced the greatest improvement in the thermal-fracture resistance. Bodies containing BeO were able to withstand as much as 70 per cent more heat flux before failure from thermal stresses than similarly prepared bodies composed of UO_2 alone. Small SiO_2 additions had an adverse effect on the high-temperature-water corrosion resistance, whereas ZrO_2 reduced the mechanical strength of UO_2 bodies.

The variation of UO_2 thermal conductivity with temperature is important to fuel-element design and is still in doubt. Values determined by Armour[56] are about 30 per cent lower than those determined by the Massachusetts Institute of Technology (MIT).[57] The Armour data were obtained on cracked specimens, which may explain the discrepancy. Hanford[58] has made a comparison on paper of the thermal conductivity of UO_2 in gas-filled swaged fuel rods and sintered UO_2 and shows how the discontinuous UO_2 phase in the former is responsible for the lower thermal conductivity in this type of UO_2 fuel rod. The effect of radiation on UO_2 thermal conductivity is unknown. Since radiation damage to the UO_2 lattice structure is expected to anneal out at reactor temperatures, disorder caused by the accumulation of foreign fission-product atoms in the UO_2 lattice may be the chief cause of thermal-conductivity changes in irradiated UO_2. Doping UO_2 bodies with selected impurities may simulate this fission-product effect, and thermal-conductivity studies on this material may show how sensitive the thermal conductivity is to impurities.

Two other thermal properties of interest to the reactor technology of this fuel are thermal expansion and melting point. For UO_2 compacts having 65 per cent theoretical density under thermal conditions simulating reactor power surges, KAPL[59] reports thermal expansion values of 2×10^{-6} to 10×10^{-6}/°C. Large UO_2 volume expansions from 1 to 7 per cent have been reported[44] for center-line melting in the Pressurized Water Reactor (PWR) rods. Since center-line melting was unexpected in several of these irradiations, thermal properties of PWR fuel rods are being re-examined. The melting point of UO_2 is one of these. A value of 2750 ± 40°C has been reported[60] and compared with previous literature values. In this study no effects of argon, hydrogen, and helium atmospheres on the melting point of UO_2 were observed.

Battelle[3] is studying the effects of nitrogen impurities on the electric conductivity of UO_2.

The chemical properties of UO_2 pertinent to reactor technology continue to be of great interest. The zirconium-UO_2 reaction has been studied at various temperatures.[61] Between 750 and 1200°F, the reaction rate is very slow. Readily measurable reaction zones were produced in about one month at 1300°F and in a few hours at 1600 and 2000°F. Zircaloy-2 apparently reacts less rapidly with UO_2 than zirconium. The oxidation of UO_2 and the changes in lattice dimensions which occur are also of interest. The dry-air oxidation mechanism is being investigated.[21] There is some evidence that diffusion of oxygen is the rate-controlling step in

the oxygen to uranium ratio range 2 to 2.34. Since crystal changes following air oxidation of UO_2 to higher oxides fragment this material, the program[26] to stabilize the fluorite structure of UO_2 during oxidation by compensating the valence change from 4 to 6 with divalent and trivalent oxides is interesting. Diffusion processes, of course, play an important role in all solid-state phenomena of interest to UO_2 technology. Diffusion studies of quadrivalent uranium and oxygen in UO_2 have been described. For quadrivalent uranium an activation energy of 120 kcal/mole has been reported[44] for self-diffusion at 1560 to 1735°C. Coefficients for self-diffusion of oxygen have been reported[21] as follows:

Temp., °C	Diffusion coefficient, cm^2/sec
395–650	4.6×10^{-6} exp (−35,500/RT)
450–600	2.6×10^{-5} exp (−29,700/RT)

(W. S. Diethorn)

Radiation Effects in Uranium Dioxide

Bettis, Hanford,[48] and KAPL are sources of radiation-effects data on UO_2. In a cooperative program with KAPL, BMI has conducted post-irradiation heat-treatments of UO_2 annularly loaded pins to determine fission-gas release rates. Detailed radiation-effects data are available from the Pressurized Water Reactor Program.[44–46] Uranium dioxide is very stable to radiation, the open nature of the fluorite structure accommodating fission products with very little distortion of the lattice.[44] Typical fission-gas release data are given in Table I-10.

Table I-10 POSTIRRADIATION HEAT-TREATMENT* OF UO_2

Theoretical density, %	Temp., °C	Heating time, hr	Kr^{85} released, % of total
96.7	1400	4	2
85.3	1600	4	50
89.9	1400	4	22
75.2	1400	4	24
80.0	600	5	0.027
91.0	600	6	0.073

* Data from Bettis Plant, Westinghouse Electric Corp., Pittsburgh, Pa.[45]

Irradiation of PWR rods to uranium burn-ups as high as 30,000 Mwd/ton show that very little, if any, UO_2 dimensional change occurs. Accordingly, most PWR rods show only small dimensional changes. When center-line melting occurs and cladding temperatures are very high, cladding strength is severely reduced, and the rod is distorted by fission-gas pressure. In general, changes in UO_2 microstructure that occur during irradiation can be attributed to temperature effects only. Work on UO_2 annularly loaded pins at KAPL seems to support the Bettis observation. Most of the UO_2 pellets in the PWR rods fracture during irradiation, but there is apparently no evidence that factors other than thermal stress are responsible for this.

Fission-gas release from UO_2 bodies has been studied over a wide range of density, burn-up, and temperature. The burn-up effect is small, whereas the effects of density and temperature on the rate of fission-gas release are large. There is evidence that fission gas is released by a solid-state diffusion mechanism. Rates are a strong function of the amount of open porosity in a UO_2 body. At theoretical densities greater than about 92 per cent, open porosity is very small, and fission-gas release rates are at least an order of magnitude smaller than those for lower densities. Of the total amount of fission gas produced in irradiated PWR rods, 1 to 10 per cent, depending on UO_2 density and temperature, is released during irradiation.

When partial melting of UO_2 occurs during irradiation of these rods, 15 to 20 per cent of the fission gas is released. Postirradiation heat-treatments release much smaller (by a factor of 10) amounts of fission gas.

(W. S. Diethorn)

Graphite as a Fuel-element Matrix

Several methods for fabrication of fueled-graphite specimens on a laboratory scale have been known for some time.[62] Currently, Great Lakes Carbon Corp., National Carbon Co., and Speer Carbon Co. are determining the feasibility of applying these techniques to fabrication of full-scale fuel elements. (W. C. Riley)

Other Ceramic Fuels

The ceramic fuels other than the UO_2 type may be divided into two classes: (1) those containing only a uranium or plutonium compound,

in which the quantity of uranium or plutonium is determined by the stoichiometric composition of the compound, and (2) those consisting of a uranium- or plutonium-bearing compound mixed with, or dissolved in, a nonfissionable phase. The demands for reactors operating at higher temperatures and the advantages thereof have created much interest in the refractory type fuels.

Development of fabrication techniques for thoria-urania fuel-core ceramics was reported by ANL.[14,63] Approximately 400 kg of cylindrical fuel pellets with a porosity of about 20 per cent were made by compacting and sintering powder batches containing 6.6 wt. % U_3O_8 mixed with ThO_2. The use of U_3O_8 rather than UO_2 as a starting material permitted sintering the ceramic compacts in air at 1800°C without disruptive volume change or significant loss of volatile uranium compounds, as determined by activity surveys of the kiln, during sintering. The report of negligible volatilization from the ThO_2-U_3O_8 compacts tends to confirm reports by NBS concerning the high stability of ThO_2-UO_2 ceramic fuels exposed to flowing hot air.

The fabrication of cermets of thoria reinforced with molybdenum, niobium, or zirconium fibers was reported by Armour and ANL.[60] Thoria ceramics, notoriously susceptible to thermal-stress failure, were improved in this characteristic by incorporating 4 to 15 wt. % of molybdenum or niobium fibers in compacts fabricated by hot-pressing. Of the three metals that were tried as fiber reinforcement, molybdenum was most effective, niobium was less so, and zirconium gave no improvement in thermal-shock resistance. The development of metal-reinforced composites may offer a means for circumventing a major shortcoming of thoria, the most refractory of ceramic oxides.

Ceramic materials containing less than 50 wt. % UO_2 have been evaluated as to their ability to retain fission gases. Bettis,[54] in comparing Al_2O_3, BeO, and ZrO_2 bodies containing UO_2 with pure UO_2 at 100°C, found that Al_2O_3 – 21 wt. % UO_2 and ZrO_2 – 12.5 wt. % CaO – 15 wt. % UO_2 released four and two times as much fission gas, respectively, as did pure UO_2. With increasing temperature the ZrO_2-CaO-UO_2 body showed the greatest increase in fission-gas release. No data are available, as yet, on the BeO-UO_2 bodies. Oak Ridge[52] is investigating ThO_2-UO_2 bodies and siliconized SiC-UO_2. Of the total Xe^{135} produced in the ThO_2-UO_2 body, 0.2 wt. % was released during high-temperature (1000°C) irradiation, compared to the release of only 0.001 wt. % of that produced in the siliconized SiC-UO_2 body. The latter material irradiated at low temperature and then heated to 1000°C lost 0.02 wt. % of the total Xe^{135} produced. Knolls[64] has initiated a program to evaluate Zircaloy-2 clad Al_2O_3-UO_2 fuel.

Fabrication of dense, spherical particles of UC or UZr by arc fusion was reported.[64] The spherical particles are useful for radiation-damage studies on alloy claddings or matrices in which the ceramic fuels are embedded. Also the spherical shape might facilitate the rolling or forging of metallic-matrix fuel-element cores.

Studies of the fabrication of UC–Zircaloy-2 cermets by Armour[65] included experiments on hot pressing, cold pressing and sintering, warm pressing, extrusion, and hot rolling. Hot pressing at 1200°C gave best results, producing pellets of near-theoretical density from compositions containing up to 50 wt. % UC. Rapid diffusion of UC in Zircaloy-2, occurring at temperatures above about 1000°C, limited the range of feasible fabrication techniques and the UC content of the cermets. Hot-pressed compacts with more than 30 wt. % UC were not resistant to corrosion in water at 680°F; areas of the cladding metal that were contaminated with diffused uranium corroded rapidly.

Table I-11 SINTERED URANIUM COMPOUNDS*

Compound	Sintering atmosphere	Sintering temp., °C	Sintered bulk density G/cm³	Sintered bulk density Per cent of theoret.
U_3Si_2	Vacuum	1470	12.05	98.6
USi	Vacuum	1500	9.89	95.0
USi_2	Vacuum	1560	8.98	100.0
USi_3	Vacuum	1500	7.58	93.0
UAl_2	Vacuum	1350	7.16	88.0
UBe_{13}	Argon	1500	3.93	90.0
UN	Vacuum	1850	11.83	82.5
UB_2	Vacuum	1700	7.6	59.0
UC_2	Vacuum	1850	10.89	93.5

* Data from Battelle Memorial Institute, Columbus, Ohio.[3,26,68]

Experiments on compound preparation and fabrication of UC_2, UB_2, UN, UAl_2, UAl_3, U_3Si, U_3Si_2, USi, USi_2, USi_3, and UBe_{13} were reported by BMI.[3,27] The compounds, prepared by arc fusion, solid-solid reaction, or solid-gas reac-

tion, were crushed to powder, compacted isostatically at 100,000 psi pressure, and then sintered in vacuum or argon at 1175 to 2300°C. The sintering conditions and maximum bulk densities obtained on the compounds that were successfully fabricated by this method are given in Table I-11.

Reactions of sintered specimens of the silicides (U_3Si_2, USi, USi_2, and USi_3), UAl_2, and UC_2 with oxygen and nitrogen were investigated at 300 and 500°C, and the parabolic rate constants were reported.[66,67] Thermal-expansion data on the silicides and the carbides are given. The oxidation of UBe_{13} in dry air at 2300°F is reported[26] to be 5 mg/(cm²)(hr). Radiation-damage experiments on UBe_{13} have been initiated at KAPL, with no results available as yet.

(J. F. Quirk and D. A. Vaughan)

Dispersion Fuel Materials

This section of the review is concerned only with dispersion fuel materials prepared by powder-metallurgy techniques, as distinguished from fuel in which the uranium is concentrated in a dispersion phase formed during cooling or subsequent working and heat-treatment of a cast alloy.

At the present time the most popular and perhaps the most versatile dispersion fuel material, as defined here, is the stainless-steel clad stainless steel–UO_2 system, in which the steel is an austenitic variety and the oxide loading constitutes between 20 and 40 wt. % of the core. During the past few months, several reports have been issued pertinent to the fabrication and irradiation characteristics of this type of fuel material.

A recent Sylvania report evaluates the effect of three major processing variables on the oxide breakup, extent of stringering, and gas permeability of stainless steel–UO_2 fuel pins prepared by a cold-compacting, sintering, coining, hot-swaging, and cold-drawing process. The variables investigated included oxide particle size (at a constant loading of 30 wt. %), sintered density of core compacts, and swaging temperatures. In general, it was found that a fine UO_2 particle size, less than 44 μ, resulted in the most random UO_2 distribution. A swaging temperature of 1200°C was optimum. As-sintered core density had no effect on the final density, but, as the sintered density was increased, there was less tendency for the oxide particles to be broken up during fabrication.

Similar results were obtained at Battelle in the development of stainless steel–UO_2 fuel elements. Plate elements prepared with pressed and sintered core compacts with densities of 77 per cent of theoretical contained a core with badly stringered and fractured UO_2 particles. However, elements in which the core compacts were recoined to a density of 87 per cent of theoretical had a much improved core structure. Other reports[73] follow in detail the development, testing, and evaluation of this type of element. Early work[23] was concerned with preparing 0.060-in.-thick elements of type 347 stainless steel, fabricating these plates into 1-in.-square by 27-in.-long subassemblies for in-pile loop testing, and evaluating their resistance to radiation damage at 1500°F in a series of capsule tests in the MTR. The emphasis has changed in more recent work, in that a 0.045-in.-thick flat-plate element fabricated from type 318 stainless steel with 25 wt. % UO_2 in the core is being developed for subsequent forming into a concentric tube array. The necessity for high strength and radiation stability at elevated temperatures, plus a high degree of formability, will require a dispersion element possessing optimum properties.

Within recent months the Army Package Power Reactor (APPR) achieved criticality. The detailed specifications for the fabrication of the stainless-steel fuel components are presented in an Oak Ridge report.[69] Included are methods for preparing uranium dioxide–boron carbide–stainless-steel cores, techniques for roll-bonding this material into composite plates, procedures for brazing these plates into flat-plate fuel components, and the finishing operations required to prepare the assembly for loading into the reactor core.

Two recent and independent literature surveys have been made by Bettis[74] and BMI on the radiation stability of the stainless steel–UO_2 dispersion element. Both sources clearly demonstrate the decrease in permissible burn-ups with increasing temperatures of irradiation. For example, dispersion elements containing 25 to 30 wt. % UO_2 (highly enriched) have withstood over 50 per cent burn-up of the U^{235} atoms at temperatures up to 600°F. However, at temperatures of approximately 850, 1500, and 1900°F, failures have occurred in similar elements at U^{235} burn-ups of 27, 8, and 3 per cent,

respectively. As would be expected, pin type elements have generally failed by longitudinal cracking through the clad, whereas plate elements have failed by blistering of the clad and core. It is discouraging to note, however, that after such a large number of capsule tests, practically no data are available on the extent of diffusion of the gaseous fission products through the stainless-steel cladding.

Other dispersion work[71] has been concerned with investigating UN and UC dispersions in stainless steel in an effort to take advantage of the higher density and uranium concentration of these fuel materials as compared with UO_2. The work with UN has been particularly encouraging, in that core structures significantly better than those obtained with UO_2 can be produced using identical fabrication procedures. This is attributed primarily to the higher strength of the UN particles. UC dispersions are also obtainable; however, fabrication temperatures must be restricted to 1900°F and lower to prevent reaction between the fuel particles and the stainless-steel matrix. Present activities on this program involve fabrication and encapsulation of UN and UC dispersions for high-temperature irradiation tests.[26]

Work on aluminum-base dispersions is continuing at Oak Ridge and at Sylvania-Corning Nuclear Corp. Sylvania-Corning[21] is investigating the extent of reaction between aluminum and various grades of UO_2 at temperatures encountered in the fabrication of roll-clad fuel elements. Specimens containing 54 wt. % UO_2 were roll clad at 600°C with a total reduction of 75 per cent. Samples subsequently heat-treated at 600°C showed a strong influence of UO_2 type and particle size on the extent of reaction. Gross distortion was observed after 2 hr at 600°C with the fine (−325 mesh) oxide.

Dispersions containing 29 vol. % U_3O_8 (−100, +325 mesh) were fabricated at 600°C and heat-treated for as long as 504 hr at 600°C by Oak Ridge.[10] After 160 hr, no significant distortion was measured; however, conversion of U_3O_8 was reported to be 25 per cent complete as determined by X-ray analysis. No uranium-aluminum compounds were detected. Plates containing −325 mesh U_3O_8 showed no measurable change in dimensions after 64 hr at 600°C; however, a 0.3 per cent increase in volume occurred after 160 hr.

In work on aluminum-UC dispersions, Sylvania-Corning[21] reports that stoichiometric UC appears completely compatible with aluminum at 600°C, whereas Oak Ridge results[10] with compacts of uranium carbide in aluminum disagree. These are summarized below.

Carbon content in uranium carbides, wt. %	Results
4.46	Disintegration in 10 hr at 600°C
4.86(UC)	87.6 vol. % increase in 10 hr at 600°C
5.75	70.6 vol. % increase in 10 hr at 600°C
6.96	4.6 vol. % increase in 10 hr at 600°C
7.98	0.6 vol. % increase in 10 hr at 600°C
8.24	No change in volume after 96 hr at 600°C
9.20(~ UC_2)	No change in volume after 96 hr at 600°C

A program directed toward providing a better understanding of the effects of structure and composition on the radiation stability of zirconium-base fuels is under way at KAPL.[68,72] During this program, dense particles of uranium, UO_2, UC, and UZr_2 will be irradiated and evaluated as powders. Similar fuel particles, unirradiated, will then be incorporated into matrices of Zircaloy-3 and zirconium-uranium terminal solid-solution alloy and irradiated. Results will be compared with wrought uranium-zirconium alloy fabricated from cast material. Hot extrusion will be used for fabricating these alloys. Data on the extrusion of both uranium shot and UO_2 dispersion in Zircaloy-3 in the range of 670 to 770°C are reported.

A unique dispersion fuel material consisting of discrete uranium particles surrounded by a magnesium matrix has been under study at Hanford[21] and earlier at KAPL. Uranium punchings, machine testings, and metal shot have been incorporated in both pure magnesium and a magnesium-silicon alloy. Loadings of 65 vol. % uranium have been successfully fabricated and canned in aluminum and in Zircaloy-3. Specimens, 0.40 in. in diameter canned in 0.050-in.-wall Zircaloy-3 and containing 50 vol. % uranium, were irradiated to 20,000 Mwd/ton (2.32 at. % burn-up in the uranium) without dimensional changes in the Zircaloy-3 can and only

slight changes in ductility and strength of the core.[21] However, similar materials fabricated at KAPL[21] with a 0.136-in.-wall aluminum can showed expansions as great as 4.41 and 10.2 vol. % after exposures of only 1000 and 3000 Mwd/ton, respectively. (D. L. Keller)

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Graphite

Determination of graphite stored energy from samples[1] exposed in the Materials Testing Reactor (MTR) for 10,000 Mwd/ton at 650°C indicated that about 8.9 cal/g could be released by an activation energy of 104 kcal/gram-atom (3 hr thermal anneal at 1000°C). Samples exposed at Hanford[2] for 4500 Mwd/ton at 400°C required a heat-treatment equivalent to an activation energy of only about 90 kcal/gram-atom (3 hr at 800°C) for complete annealing of 13.4 cal/g of stored energy. These data indicate that graphite irradiated at high temperature for extended periods will not build up sufficient stored energy to cause reactor operating difficulties.

Nuclear purity tests[1] indicate that (1) GBF graphite processed in 8- by 8-in. cross sections is as pure as 4- by 4-in. cross-section material and (2) resin impregnation of either AGOT or GBF graphite, with subsequent heat-treatment to 900°C, results in sufficient impurities to prevent their use as massive moderators for plutonium-production reactors.

High-density graphite is required for the moderator of a proposed liquid-metal-fuel gas-cooled reactor.[3] Coating on the graphite, impregnation, or an impervious coolant-passage lining will be required to prevent diffusion of the helium coolant and fission gases. Techniques for accomplishing these, together with chemical-reactivity studies of graphite in its environment, are subjects that require investigation.

(W. C. Riley)

Beryllium

Beryllium Metal and Alloys

Research on the metallurgy of beryllium continues to be concerned with three main areas of work: (1) studies of the brittleness problem, (2) studies on the high-temperature strength of alloys containing a dispersed intermetallic phase, and (3) studies of corrosion and oxidation resistance in various mediums.

Various fundamental and practical aspects of the brittleness problem in beryllium are under study. Workers at Nuclear Metals[4,5] are continuing the basic studies on the crystallography of flow and fracture of beryllium begun at MIT. That work showed that beryllium single crystals (hexagonal close-packed structure) slip on both basal (0 0 0 1) and type I prism planes $\{1\,0\,\bar{1}\,0\}$. At room temperature the critical resolved shear stress for basal slip is about one-fifth that required for prismatic slip. Fracture in beryllium single crystals occurs on basal planes (0 0 0 1) and on type II prism planes $\{1\,1\,\bar{2}\,0\}$. Unique values for the fracture stress on these planes do not appear to exist but seem to depend on the mode of deformation preceding fracture.

The most desirable crystal orientation for ductility in extruded powder-beryllium rod (in the direction of extrusion) results from having the c axes of the individual crystallites perpendicular to the direction of the rod. In sheet products, the best ductility (in the plane of the sheet) is obtained by having the basal planes lie in the plane of the sheet. Under these conditions basal-plane fracture is suppressed, and $(1\,1\,\bar{2}\,0)$ fracture determines the ductility in the direction of the extruded rod or in the plane of the sheet, as the case may be. The current Nuclear Metals work is concerned with studying the effect of alloying additions on $(1\,1\,\bar{2}\,0)$ fracture. Single-phase binary alloys containing up to 5 wt. % of the elements chromium, copper, iron, and nickel are being studied. The alloys are being fabricated (by extrusion and rolling of powders) such that the basal (0 0 0 1) planes are in the plane of the sheet in order to suppress basal fracture, as noted previously. Basic data of this type will be helpful in understanding the effect of alloying additions on the flow and fracture behavior of beryllium.

Workers at KAPL[6] have been concerned with producing cast beryllium ingots with fine equiaxed grains. Using beryllium – 2.5 wt. % aluminum as a base alloy, they found that additions of SiC, CrB_2, Cr_3C_2, BN, and BeO (added in the mold prior to pouring) were effective in refining the as-cast grain size. They noted that aluminum and silicon appeared to be the only alloying elements that tend to give an equiaxed grain structure in cast beryllium. Rolling studies

are being conducted on these fine-grained alloys. It would be desirable to produce cast and fabricated beryllium with properties equal to those of the extruded-powder products. At the present time the best combination of strength and ductility is exhibited by the latter.

The strength and ductility of bolted and brazed joints of beryllium has been the subject of studies at Nuclear Metals.[7,8] The fracture strength of bolted joints in beryllium sheet had been found to be only one-fourth that of the metal itself. Annealing of the ultrasonically drilled sheet at 750°C for 18 hr was found to increase the strength and ductility of the bolted assembly to the following values: ultimate tensile strength, 86,500 psi; strain at ultimate, 20.6 per cent. In a study of brazed lap joints of beryllium sheets using an aluminum-silicon braze metal, it was found that fracture occurred at relatively low stresses (10,000 to 20,000 psi). Generous filleting of the steps of the lap joints with braze metal was found to approximately double the aforementioned values of fracture stress. The braze metal per se was not held responsible for the embrittlement, since a conventional beryllium-sheet tensile specimen coated with the brazing alloy was not embrittled in comparison with an uncoated specimen. It was believed that the stress concentration existing at the steps of the lap joints contributed to the embrittlement. The work appears to indicate that metal-joining techniques can be satisfactorily applied to beryllium if care is taken to minimize stress concentrations.

The research to develop beryllium alloys with high-temperature strength is largely concerned with producing alloys containing dispersed phases of various intermetallic compounds in beryllium. Studies[4,9] are being made on the effect of additions of SiO_2, Fe_2O_3, and Al_2O_3 on stress-rupture properties. Data for the 3 and 6 vol. % SiO_2 alloys indicated no improvement in the stress-rupture properties. Brush Beryllium Co.[10] is studying the effect of 1.4 to 16.7 wt. % $FeBe_{11}$ on the high-temperature properties of beryllium. Alloys are being prepared from elemental-beryllium and -iron powders; one of the processing variables is the particle size of the iron. Results of this study are not yet available. Other data show the effect of BeO contents of 1 to 3 wt. % on the tensile and stress-rupture properties of beryllium at 1000 to 1350°F. The tensile yield strength increased from approximately 12,000 to 21,000 psi over the range of BeO content indicated above. It should be noted that a BeO content of about 1 wt. % is to be expected as a base oxygen level in extruded-powder products of beryllium.

Some data from MIT[11] showing the effect of about 2.5 wt. % Be_2C on the stress-rupture life of beryllium at 1200 to 1350°F are presented in Table II-1. The increase in stress-rupture life at 1200 to 1350°F resulting from the Be_2C addition appears especially promising.

Table II-1 STRESS-RUPTURE PROPERTIES OF Be-Be_2C EXTRUDED-POWDER PRODUCT*

Material	Temp., °F	Stress, psi	Rupture life, hr
Unalloyed Be	1,200	10,000	0.2
Unalloyed Be	1,200	5,000	1.6
Be–2.5 wt. % Be_2C	1,200	10,000	4.0
Be–2.5 wt. % Be_2C	1,200	5,000	181.0
Unalloyed Be	1,350	2,000	0.95
Be–2.5 wt. % Be_2C	1,350	3,000	860.0

* Data from Massachusetts Institute of Technology, Cambridge, Mass.[11]

Current research on corrosion of beryllium is concerned with obtaining specific corrosion-rate data in air and in various heat-transfer mediums at elevated temperature. At Nuclear Metals[4,9] binary alloys containing small amounts of aluminum, iron, nickel, silicon, titanium, and zirconium, as well as unalloyed beryllium, are being studied. No significant weight change was observed for any of these alloys after 516 hr exposure at 600°C in still air. Tests in 600°F water on binary alloys containing up to 0.53 wt. % chromium, 0.046 wt. % iron, or 0.47 wt. % nickel produced weight gains of from 46 to 335 mg/dm^2 after two weeks of exposure. The attack was local but was believed to be associated with porosity in the powder-metallurgy samples rather than with alloy segregation. The weight gains were greater than those observed for unalloyed beryllium (10 to 20 mg/dm^2). In the Brush Beryllium studies with 600°F deionized water, the effects of surface condition (turned, ground, or chemically polished) were evaluated. The corrosion rates for the first 14-day period were about 40.5 mg/(dm^2)(month) for the turned surface and about 44,000 mg/(dm^2)(month) for the chemically polished surface.

The corrosion of beryllium in low-oxygen sodium (reactor grade, 10 ppm) is being studied by North American.[12] Some rate data obtained on

2- by 5- by 0.11-in. sheet samples are reproduced in the following tabulation.

Conditions	Test temp., °F	Exposure time, hr	Weight change, mg/cm²
Static test	950	260	+0.11
	950	500	+0.09
	950	1024	−0.12
Dynamic loop test	1000	507	+1.7

(A. J. Griest)

Beryllium Oxide

The recent reports on work with BeO describe experiments on compatibility of BeO with stabilized uranium oxide ceramic fuels, fabrication of high-temperature fuel-element ceramics made from sinterable BeO, characterization of sinterable BeO powder, and compressive creep of BeO ceramics.

Studies on the compatibility of BeO and other refractory oxides with stabilized ceramic fuel compositions were reported by NBS. One of the best of the stabilized oxide fuels, a UO_2-ThO_2 solid solution, was found to be compatible with BeO. No deleterious reactions were observed between UO_2-ThO_2 fuel grains and BeO when tested in oxidizing gas at 1800°C.

(J. F. Quirk)

Solid Hydrides

Calcium, niobium, and zirconium hydrides have all been given consideration as possible moderators for power reactors, but, of these, the material receiving by far the most attention has been zirconium hydride. The principal objectives of the various hydride-moderator development programs have been (1) the determination of the physical, chemical, and mechanical properties of the materials and (2) the investigation of methods for protecting them.

Calcium Hydride

Recent research on calcium hydride has been limited to studies at BMI[13] of its thermal conductivity and its compatibility with various metallic cladding materials. The thermal conductivity of a compacted powder specimen of calcium hydride was found to range from 0.500 to 0.633 Btu/(ft)(hr)(°F) between 600 and 1600°F. The compatibility tests showed that copper, nickel, and Nichrome V all diffuse extensively into calcium hydride at 1700°F and are, therefore, unsuitable cladding materials under these conditions. Molybdenum, chromium, niobium, and type 446 stainless steel are compatible with calcium hydride at 1700°F.

Niobium Hydride

Recent studies of the phase equilibriums in the niobium-hydrogen system[14] have shown that niobium does not form a true hydride but takes in hydrogen in solid solution up to a limiting hydrogen to niobium atomic ratio of about 0.85. Earlier work at Sylvania[15] had indicated a limiting ratio of 1.1. The relatively low solubility of hydrogen in niobium and the relatively high dissociation pressures of these solid solutions limit the use of niobium hydride as a moderator to large reactors operating at temperatures below about 800°F.

Zirconium-Uranium Hydride

Some consideration has been given by BMI,[14] Sylvania,[16] Bettis,[17] and Westinghouse Research[18,19] to the use of the hydride of a zirconium-uranium alloy as a combined moderator and fuel. Uranium additions varying from about 1 to 50 wt. % have been studied. The practicality of such a combination has not yet been demonstrated. It has been shown, however, that the uranium separates out from the alloy upon hydriding and thermal cycling, but it is not known to what extent this affects the utility of the material.

(J. B. Vetrano)

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Control-rod Alloys

Silver-Indium-Cadmium Alloys

A silver–15 wt. % indium–5 wt. % cadmium alloy appears to be a promising control-rod material. As a neutron absorber it compares with hafnium and is superior to a silver–30 wt. % cadmium alloy. During exposure to high-temperature water, a tightly adhering corrosion film is formed which should prevent the introduction of induced radioactivities into the primary coolant.[1] The alloy showed no adverse visual effects nor dimensional changes after irradiation.[2] The effect of irradiation on mechanical properties has not been investigated.

The effect of small additions (probably <1 wt. %) of platinum on the strength of the alloy has been studied. The yield strength increased from 13,100 to 34,800 psi, with a negligible decrease in ductility. Alloys of silver–15 wt. % indium–5 wt. % cadmium and silver–15 wt. % indium–5 wt. % cadmium–0.5 wt. % aluminum, produced by powder-metallurgy techniques, had higher yield strength and ultimate strength than solid-metal extrusions. These powder-metallurgy materials had increased resistance to softening and grain growth upon annealing. This is probably the result of microscopic oxides or some other impurity picked up during compacting.[2]

Boron Alloys

Alloys of Zircaloy-2, with up to 0.35 wt.% B^{10}, were investigated. Little difficulty was met in casting, fabricating, or cladding these alloys. However, they were severely damaged upon being irradiated. For example, irradiation to 90 per cent poison atom burn-up, in a thermal flux of 2×10^{14} nv, resulted in a large number of thin, longitudinal cracks over the entire length of the elements. Damage is apt to be large in boron materials because B^{10} captures neutrons by an n,α reaction, so that two foreign atoms are produced in the material for every neutron captured.

Rare-earth Alloys

Homogeneous castings of up to 25 wt. % gadolinium in stainless steel, titanium, and zirconium have been produced without much difficulty using standard arc-melting techniques. Gadolinium–stainless steel alloys were successfully formed by forging and rolling operations. A program to study alloys containing europium and samarium is planned.[3] (V. W. Storhok)

Dispersion-control Materials

Irradiation for five reactor cycles has been conducted on a specimen of 5 wt. % $B_2^{10}O_3$ in Vycor.[4] Helium analysis is reported. The helium was released without deformation of the Vycor. The specimen temperature was estimated to be 700°C, and cracking was observed.

A separate report[5] gives the effects of irradiation on the properties of boron–stainless steel powder dispersions. Specimens containing 1 wt. % B^{10}, 2 wt. % B^{10}, and 5 wt. % natural boron were irradiated to unperturbed exposures of 1×10^{21} nvt. Although no gross dimensional change nor warpage was produced, tensile and impact tests indicated that the specimens were completely embrittled (hardness increased as much as 175 DPH) as a result of the irradiation.

Oak Ridge specifications have been released for the fabrication of reference control rods for the APPR.[6] The rod consists of a dispersion of enriched boron in iron, clad with type 304L stainless steel.

In addition to the use of boron in control rods, dispersions of boron have also been prepared for use as burnable-poison elements. A discrete zirconium burnable-poison element was fabricated successfully at Knolls; however, because of damage incurred as a result of irradiation, it is not considered usable. The results of pre- and postirradiation tests as well as conclusions drawn and recommendations for future investigations are given. Specimens containing 1.2 wt. % natural boron or 0.35 wt. % B^{10} dispersed in zirconium and clad with Zircaloy were irradiated to 50 per cent poison depletion. Knolls progress

reports also indicate that a ceramic strip of B_4C clad with Zircaloy is being investigated as a burnable-poison element.

Dispersions of rare-earth oxides have also been considered as control materials. The sinterability of the pure oxides of europium, samarium, gadolinium, and dysprosium has been investigated.[7] Data are presented on density, grain size and microhardness. An extrusion method of producing stainless-clad model control rods with cores of stainless steel containing dispersed rare-earth oxides has been developed.[8] Core materials consisted of 25, 30, and 40 vol.% Sm_2O_3. Detailed information is given on the round-die single hot coextrusion method for producing Y-shaped rods. Postirradiation data have been reported for dispersions of 20, 25, and 30 wt. % Eu_2O_3 in stainless steel. Bend-test data after exposure in the MTR for two to five cycles are reported. Comparison of pre- and postirradiation deflections before fracture indicates that ductility is reduced and strength is increased. The change in ductility varies over wide limits, but the ductility is decreased as the Eu_2O_3 loading is increased.

Battelle has prepared flat plate rare-earth oxide dispersions by roll-cladding techniques. Preliminary work involved the use of a Sm_2O_3-Gd_2O_3 mixture dispersed in Stellite 25, cobalt, Nichrome V, and stainless-steel matrices. Claddings used were Stellite 25, Nichrome V, and stainless steel. Dispersions of Eu_2O_3 and Gd_2O_3 in Nichrome V, and clad with Nichrome V, were also prepared. Specimens were roll-clad at 2000°F and are being evaluated by means of microstructures and hardness values.

(G. W. Cunningham)

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Melting

Developmental efforts in the melting and casting of fuel materials for reactors have been centered around metal quality and alloy homogeneity. National Lead Co. of Ohio, in studies of the melting of pure uranium at their Feed Materials Production Center (FMPC) at Fernald, Ohio, have shown that control of vacuum-melted ingot chemistry can be related to the charge make-up.[1] The elements carbon and nitrogen were particularly noted. Carbon content was lowest (400 to 500 ppm) for ingots cast from chipped derby charges. Ingots from briquette-chip charges were, on the average, 90 ppm higher, and ingots from charges of pickled derbies, reject slugs, or rolling-mill scrap averaged 200 ppm higher in carbon than the chipped derby charges. Nitrogen was found highest in ingots cast from chipped derby charges, approximately 70 ppm. Briquette-charge ingots had the lowest nitrogen content (26 ppm). The ingots from the other types of charges, rolling-mill scrap, pickled derbies, and reject slugs, had nitrogen contents of approximately 45 ppm. In associated studies the yield of good slugs was found to be lowest for ingots melted from charges made up completely of rolling-mill scrap. A patent of recent issuance also of interest in the melting of uranium was assigned to Spedding and Wilhelm of Ames.[2] The patent refers to melting of uranium in carbon crucibles and claims that excessive carbon pickup is avoided by the formation of a thin insoluble layer of UC on the crucible wall. The protection is said to be effective for melt temperatures up to 1650°C.

A theoretical study of the variables involved in the solidification of uranium ingots has been initiated at BMI.[3] The objective of the work is to obtain information on the control possible in casting of uranium ingots. Mathematical models of heat flow during solidification will be subjected to electronic-computer analysis.

The preparation of pure-uranium shot has been the subject of a successful developmental study by National Lead. They report that total yields above 90 per cent are possible.[1] High yields in the small-size fractions (−100 mesh) were obtained by pouring metal onto a high-speed spinning refractory disk. The enclosure of the melt and disk in a 12-ft-diameter tank containing 75 mm Hg pressure of helium prevented oxidation and provided the cooling required for the production of near-spherical particles.

Several other studies involving aspects of unusual melting and casting procedures are under way. In this connection the Bureau of Mines reports that basic, unresolved differences exist in the metallic arc of titanium and zirconium when comparison is made with the arc produced by copper and iron.[4] A recent book,[5] *Arc in Inert Atmospheres and Vacuum*, is devoted entirely to the electric arc. Topics ranging from the historical to the chemical applications are covered. This book is suggested as a ready reference for those interested in arc melting.

The Bureau of Mines is also conducting a study to determine the practicability of cooling arc-melting crucibles with air.[4] The results to date appear promising. The impetus for this study was the occurrence of several explosive accidents that resulted when the crucible burned through and the cooling water contracted the molten titanium. Another study conducted by the Bureau of Mines pertains to the centrifugal casting (rotating mold with axis vertical) of tubular zirconium shapes.[4] The product of this development is being considered as the starting material for extrusion-cladding operations. Feasibility has been established.

Nuclear Metals is producing rods of uranium–10 wt. % niobium alloy by compacting machine chips. Excellent homogeneity is reported, but questionable corrosion properties were exhibited.[6] In an effort to reduce the carbon content of uranium-niobium alloy melted in graphite, small amounts of zirconium were added to the melt. Some flotation of the carbon as ZrC was noted for quiet melts.[7] Attempts are also under way to reduce segregation during solidification by agitation through induction stirring in the mold. (E. L. Foster, Jr.)

Extrusion and Forging

Pure uranium shapes, 10 in. diameter (pilot ingots from National Lead and Mallinckrodt Chemical Works (MCW) dingot) were successfully forged to 7-in. bar at MCW.[8] The forged material is to be rolled at FMPC in the same manner as National Lead's 7-in.-diameter ingots.

Efforts at MCW on the extrusion of dingot shapes are principally concerned with the evaluation of lubricants.[8] Best rod yields were obtained using glass lubricants at 1900°F with graphite follower blocks. Lower extrusion pressure was required, and die life was extended to 30 billets with glass lubrication, as compared with 3 billets for a graphite suspension type lubricant.

The production of uranium washers that are assembled to produce cored fuel slugs is the concern of National Lead.[1] They found that hot (1100°F) impact forging small solid blanks, followed by machining, has increased metal yields 25 to 35 per cent and made possible substantial savings when compared with the present method of cold punching sheet.

Bridgeport Brass is actively engaged in the development of procedures for the extrusion of Hanford cored slugs. The remaining problems are the eccentricity of the core and coarse grain size of areas near the core. Despite continuing efforts yields of only 80 per cent are frequent. The occurrence of coarse grains has been attributed to allotropic phase transformation occurring during the extrusion process as a result of high mandrel temperatures. Recorded mandrel temperatures indicated that the beta transformation temperature was exceeded and that higher temperatures associated with the gamma range were likely. This hypothesis has been strengthened by work that suggests a correlation between the occurrence of coarse grains and hole size in the billets.[9]

(E. L. Foster, Jr.)

Cladding

Roll Cladding

At present process evaluation of roll cladding of nuclear components is receiving almost as much attention as the further development of the art of roll cladding. Production yields are being investigated in an attempt to produce components more economically by roll cladding. Development studies are being concentrated on dimensional control during rolling and on the cladding of systems with dissimilar fabrication properties.

Argonne[10] is evaluating yields of Zircaloy-clad zirconium-uranium fuel elements fabricated by roll cladding. Sylvania[11] is checking yields of roll-clad aluminum-uranium elements clad with Alcoa M-388. Yields in the range of 80 to 85 per cent of the fuel plates prepared appear to be normal. These yields are based on the material available for roll cladding and do not reflect any losses prior to the roll-cladding operation. Argonne has prepared more than 1000 Experimental Boiling Water Reactor (EBWR) fuel plates, and the number and types of rejects have been evaluated.

Some of the laboratories are using new and modified techniques previously investigated in an effort to solve the dimensional control problems involved in fuel-element fabrication. Sylvania[11] observed that increased total reductions above 2½ to 1 failed to improve bond strength significantly, whereas the increased total reduction increased the dimensional-tolerance problems.

(E. S. Hodge)

Pressure Bonding

Reactor components can be bonded by forcing the components together at high pressures in a die or with gas pressure, at elevated temperatures. This process, called pressure bonding, is currently being studied in several laboratories. In most applications of the pressure-bonding technique, the components to be clad are fabricated to final size prior to assembly.

Pressure-bonding techniques have been extensively used for cladding nickel-plated uranium with aluminum. Both uranium rod and flat-plate elements have been clad in production quantities by the hot-press die technique. Development work is also being continued on this fuel system utilizing both hot-pressing and gas-pressure-bonding techniques to study the conditions under which strong bonds are produced.

Pressure bonding is being investigated at BMI[12–14] as a means of cladding nickel-plated uranium with sponge zirconium. Pressure bonding is of interest in this application because it permits close dimensional control. Zirconium requires much higher temperatures than alu-

minum, so most of the development work on the cladding of uranium with zirconium has utilized the gas-pressure-bonding process at temperatures of 1200 to 1350°F. Bonds are metallographically sound, and some have withstood four water quenches from 1335°F before cracking occurred.

The gas-pressure-bonding process is especially applicable to the cladding of relatively brittle materials. This process is being investigated at BMI,[14] and at Bettis[15] for the bonding of Zircaloy-clad flat-plate uranium dioxide fuel elements. The oxide fuel, pressed into strips, is placed in compartmented Zircaloy frames. Covers are applied, and the assembly is sealed in a container that is subjected to gas pressure of 10,000 psi at temperatures in the vicinity of 1550°F. Bonding is achieved without deformation of the oxide cores. The quality of the bond is dependent on the method used for preparing the bonding surfaces of the Zircaloy components.

The pressure-bonding process is amenable to cladding of complex shapes and subassemblies. Battelle is studying the gas-pressure-bonding technique for the cladding and bonding of subscale Zircaloy-Zircaloy-uranium subassemblies in a one-step operation. The core, clad, and spacer components are fabricated to final size and assembled in a container that is evacuated, sealed, and then subjected to a high gas pressure at elevated temperature. Ti-Namel strips are used as spacers. After bonding, the spacer strips are dissolved to form the coolant channels. Cladding of the fuel and bonding of the fuel elements into an assembly are accomplished in one operation. Excellent bonds are produced, and close dimensional control is achieved.

A combination of gas-pressure bonding and braze annealing is being investigated by Sylvania[16] for the preparation of stainless-steel clad stainless-steel–UO_2 fuel assemblies. Multiholed wafers of stainless-UO_2 are pressed, sintered, and stacked to form an assembly. Stainless-steel tubes are passed through the holes in the wafers to form the container and ultimate cladding. The assembly is sealed and subjected to hydrostatic pressures of from 20,000 to 100,000 psi to bring all the components into intimate contact. After the pressure treatment the assemblies are either annealed at 1340°C or nickel brazed at 1120°C in a hydrogen atmosphere in order to bond the components. Since the stainless-UO_2 wafers are pressed and not deformed during the cladding operation, a uniformly dispersed cermet structure is maintained.

Battelle research[12,14] is aimed at learning the basic fundamentals of bonding. Gold needle points brought into contact with a flat gold surface are used to simulate single-asperity contacts. Bonds are produced that possess a strength that is higher than the force employed to bring the surfaces into contact. The formation of the bond and subsequent strength are dependent on the degree of cold work, time, creep, and self-diffusion at any given temperature. Other work involves an analysis and investigation of gas-pressure-bonding phenomena.[3,14] This investigation is attempting to correlate analytical studies of the process with experimental data.

(S. J. Paprocki)

Coextrusion

A special application of the extrusion process, coextrusion, has been proved as a feasible method to produce nuclear fuel elements with integral cladding and end caps. It is possible to completely envelope a core with a clad in a single operation with this technique.

The experimental and development work in coextrusion over the past few months has been directed mainly toward the improvement of the cladding at or near the core end-seal sections. A number of methods have been attempted by Nuclear Metals to reduce the nonuniformity of the cladding and to keep the length of the extrusion end-seal defect within tolerance. One of these methods involved machining matching radii on the core and end-seal surfaces.[7,17] This work was done on tubular elements consisting of a uranium–5.0 wt.% zirconium–1.5 wt.% niobium alloy core with Zircaloy-2 cladding, and on Zircaloy-clad uranium rods. This method has not been entirely satisfactory. The use of steeper angle extrusion cones, without the preshaping of the core or end-seal components, has greatly decreased the length of the extrusion defect in tubular elements containing uranium–5.0 wt. % zirconium–1.5 wt. % niobium alloy clad[18] with Zircaloy-2. Cones with angles of 60 and 75 deg were used instead of the conventional 45-deg cone. To eliminate voids between the rear end-seal to core interface of Zircaloy-2 clad uranium elements, an integral nose plug

and cutoff of a relatively stiff alloy (copper–10 wt. % nickel) to back up the cone, end seal, and cladding assembly was employed.[6] There were no voids in evidence when the stiffer alloy was used.

It may be desirable to weld end closures on the fuel rods after extrusion, since integral end closures greatly increase the extrusion cost. A method of machining, fitting, and welding to effect a corrosion-resistant closure is being investigated. One Zircaloy-clad uranium rod extrusion with 1-in.-long end plugs was produced. Metallographic examination showed some uranium contamination of the weld, but successful corrosion-resistant welds are anticipated.

Studies of cladding void defects which might be encountered during coextrusion production of Zircaloy-2 clad uranium–2 wt. % zirconium rods and which would lead to corrosion failure were conducted by Nuclear Metals.[19] Rods were produced with intentional cladding defects that were the result of either plugged or unplugged holes drilled in the cladding components before extrusion or of faulty end seals. Corrosion tests of these as-extruded rods in high-temperature water and steam showed that (1) small voids or cracks in the circumferential cladding are more likely to cause rapid corrosion failure when the extruded cladding is thin (~5 mils) than when it is thick (~30 mils), (2) if the cladding defects contain uranium metal or ZrO_2 before extrusion, corrosion failure of the extruded elements occurs readily, and (3) rods having imperfectly bonded end seals or fine threads of uranium protruding through the end seals do not exhibit rapid corrosion failure.

Basic studies have been carried out by Nuclear Metals, using Plasticene, lead, and antimony-lead alloys, in an attempt to provide an understanding of the flow of metal in a coextruded end-seal preparatory to tandem-extrusion studies.[20] The feasibility of producing 12-in.-long Zircaloy-clad uranium rod-type extrusions in tandem (6-in.-long elements) has been indicated, and work is being carried out to see whether this technique can be used for longer elements.[7,21] (C. B. Boyer)

Extrusion Cladding

An interesting study of the kinetics of layer formation at the aluminum-nickel interface in extrusion-clad nickel-plated uranium flats was made at Chalk River.[22] The objective of the work was to determine whether the rate of interdiffusion of nickel and aluminum is so rapid that the thin nickel layer (about ½ mil) will be consumed by forming diffusion layers or whether nickel will penetrate the aluminum sheath during the time the fuel element is in the reactor. It was found that, if the operating temperature during irradiation in the reactor is 175°C at the aluminum-nickel interface, the time required for the nickel layer to be completely transformed into intermediate phases is about 6½ years. For an aluminum sheath thickness of 20 mils, it would require 192 years at 300°C before the aluminum was penetrated by the diffusion layers. (R. J. Fiorentino)

Vapor-deposited Coatings

Uniform, adherent, 0.001- to 0.020-in. coatings of NbC, TaC, and ZrC were obtained at BMI[23] on both surfaces of graphite tubes by the thermal decomposition of the corresponding chlorides or bromides under conditions of pressure and temperature so chosen as to be unfavorable for the deposition of the metal but to permit formation of the carbide as rapidly as carbon diffused from the base to the surface. This principle is particularly applicable to the vapor-phase coating of tubes having a large ratio of length to diameter where normal fluid plating procedures result in a pile-up of the coating at the fluid-entrance end of the specimen. Earlier workers[24–26] have also coated graphite with carbides under empirically chosen conditions falling within the required ranges..

In coating the inside bore of stainless-steel tubing with niobium, it is not possible to use the base as one of the reactants to control the rate of coating formation as is done above. To solve the problem in this case, BMI[3,12–14,27] used a traveling heated zone to coat uniformly 0.15-in.-I.D. by 20-in. lengths of type 304 stainless-steel tubing with 0.001 to 0.002 in. of niobium by the hydrogen reduction of $NbCl_5$. (J. M. Blocher, Jr.)

Electrodeposited Coatings

The electrodeposition of about 0.018 in. of nickel improves the impact resistance of a TiC-Ni-Mo cermet up to 10-fold.[28] Little improvement was obtained by less than 0.004 in., but beyond this thickness impact resistance increased more than linearly with increasing

thickness. Only one plating process was studied, and this was slow, requiring more than 24 hr for the 0.018-in. coating. Because they found that plated coatings lacked the desired strength, Sylvania investigated vacuum-brazed coatings, infiltrated Nichrome V coatings, vacuum-cast structures, and diffusion-bonded tubing. Only the bonded tubing improved impact resistance, but results on this method were incomplete.

Werner and Katz[29] have indexed, by report number, all subjects investigated over a period of three years. Subjects include the use of a variety of techniques for the electrodeposition of metals, oxides, hydrides, carbides, or mixtures of these. (J. McCallum)

Enamels and Electrophoretic Coatings

The major problem in enameling uranium has been crazing of the coating during the cooling cycle of the firing process. To eliminate this defect, an attempt was made at BMI[3,12–14,27] to use a layer of vacuum-deposited aluminum between two enamel layers. This technique sharply reduced but did not eliminate crazing. It was also reported[13] that phosphate enamels, applied as films of less than 1 mil thickness, were highly adherent to uranium when fired in air. Minute cracks observed at the glass-metal interface did not extend to the upper surface of the coating. The tendency of such cracks to propagate under thermal cycling was not determined.

In a Vitro Laboratories report,[29] numerous references are given on ceramic or cermet coatings applied by electrophoretic deposition. This technique has the advantage that it permits the application of exceptionally uniform coatings. Use of suitable dispersing agents and binders was found to be very important in obtaining defect-free films. Coatings were applied on copper, Croloy, mild steel, stainless steel, graphite, boron carbide, Nichrome, Inconel, or nickel, using specimens of various shapes and sizes. To decrease the porosity of the coatings, they were isostatically pressed at pressures up to 100,000 psi before sintering.

A number of coatings were investigated[30] for the protection of graphite against high-temperature oxidation and erosion. These coatings, which were alloyed by electrophoretic deposition, were composed of alumina silicon carbide or one of three alloys. Although no information was given as to the value of these coatings in protecting graphite from oxidation, good wetting of the graphite was obtained with an alloy of 75 wt. % nickel, 15 wt. % chromium, and 10 wt. % silicon. It was suggested that this alloy might be used for joining graphite since good bonding was noted between the fused alloy and the graphite surface. (B. W. King)

Welding and Brazing

Development work is in progress at Hanford on two novel welding methods. Roto-arc welding is being studied for producing seal welds on aluminum-clad fuel elements. The Roto-arc consists of an inert-gas-shielded arc between a nonconsumable electrode and the aluminum can which is rotated along the weld line by a magnetic field. Low power requirements and short weld cycles are obtainable with this method. The other welding method utilizes an electron beam to produce sufficient heat for melting. The electron beam is operated in a high vacuum, which must be maintained throughout the welding cycle. Studies are being made on zirconium and zirconium alloys. The electron-beam welding method is significant because it may be a useful tool for preventing contamination of reactive metals, and it will allow the sealing of evacuated assemblies.

Many welding programs are being conducted on zirconium and Zircaloy-2 and -3. Two items are outstanding: (1) the common problem of weld-penetration variation and (2) increased interest in simpler shielding methods. Westinghouse Electric Corp.[31] has presented work to show the cause of variations in penetration which indicates that the chlorine analysis of Zircaloy must be controlled to obtain consistent penetration. Workers at KAPL[32] showed that rigid vacuum purging cycles are not necessary to maintain the corrosion properties of Zircaloy-2 and -3 weldments. However, this work and previous studies by others have not been extensive enough to establish conclusively the extent to which current specifications could be relaxed. A complete study of this area would appear to be warranted, considering the many times that the effects of atmospheric purity on corrosion properties of zirconium and the Zircaloy alloys have been studied.

Studies of the zirconium–4 wt. % beryllium brazing alloy developed at Armour are being continued at Bettis. Final development of this

or some other brazing alloy for zirconium would add a useful joining method for reactor fabrication. (R. E. Monroe)

Nondestructive Testing

As pointed out by McGonnagle et al., in a comprehensive, although general, discussion of reactor inspection needs and techniques,[33] the nondestructive testing of reactor elements requires sensitivity and resolution in excess of those that can be justified in most other industrial applications. Old methods and techniques have been improved. New techniques and specialized instruments have been and are being developed.

Ultrasonic techniques for the inspection of core materials are being developed and used at many installations. Workers at Oak Ridge[34] describe production inspection apparatus used there which will handle long lengths of tubing (down to $^3/_{16}$ in. O.D.) and pipe (up to 8 in. O.D.). They use a method of oscilloscope presentation which synchronizes the signal with the specimen rotation and permits a high-persistence easily recognizable qualitative picture of the depth and size of a defect.

However, attempts to use ultrasonic inspection for quantitative measurements of the size and type of flaw or the grain size[35] (to determine degree of metallurgical transformation) of materials have not been completely successful. The amplitude of the reflected or transmitted signal depends upon the distance of the flaw from the transmitter, the orientation and size of the flaw, and the density and elastic modulus of the flaw, as well as the density and elastic modulus of the material being inspected. Statistical correlations of inspection data with destructive examinations are being made in an attempt to establish inspection specifications and production inspection procedures.[35] However, on the basis of attempts to correlate signal pulse amplitude with size of defect, recommendations are using maximum sensitivity and rejecting all material that gives discrete echoes of any amplitude if all defects greater than 0.030 in. diameter are to be avoided.

Radiography provides a convenient and rapid means of locating the edges of core materials in clad fuel elements and inspecting for inclusions and voids in the materials and welds. In many applications it lacks sensitivity, but special techniques have increased its usefulness. Procedures developed at Bettis[36] over a period of several years provide valuable data on voltages, films, filters, exposures, and shape compensation for radiography of uranium, zirconium, and hafnium and assemblies fabricated of these materials. (W. H. Goldthwaite)

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Corrosion of Cladding Materials

Zirconium Corrosion

Of primary concern in the field of zirconium corrosion is the stringer type of attack observed for Zircaloy-2 and -3 after exposure to high-temperature water and steam. This type of corrosion behavior was first observed during the initial Zircaloy-3 evaluation program. A similar type of corrosion also has been occasionally observed for Zircaloy-2 strip coupons. No special significance, however, was attached to this observation primarily because the effect, until recently, had not been observed in tests with fabricated fuel elements.

Although the stringer type of corrosion is more often encountered with Zircaloy-3, the mechanism of the attack in both Zircaloy-2 and -3 appears to be similar. Corrosion behavior has been associated with changes in microstructure. Recent studies at Bettis[1] have shown that structures composed of a continuous network of a second phase (thought to be intermetallic compounds of zirconium with iron and nickel) at the grain boundaries are susceptible to attack, whereas those having a discontinuous second phase are resistant. A similar relation between microstructure and corrosion behavior of binary zirconium-nickel and zirconium-iron alloys was also observed in studies at BMI.[2]

In the studies at both Bettis and BMI, it was found that corrosion-resistant structures could be obtained by proper heat-treatment. Annealing treatments at 1800°F for 4 to 8 hr, followed by an air quench, have been found to eliminate stringer attack of the Zircaloys. The rate of cooling from the annealing temperature is quite critical. If cooling is not rapid, the second phase will segregate in clusters, giving rise to increased attack in these areas.

Recent studies of a more fundamental nature which have contributed to an understanding of the oxidation and corrosion behavior of zirconium and its alloys in high-temperature water and other mediums are those of Nuclear Metals,[3] Case Inst. of Tech.,[4] and BMI.[5] Nuclear Metals was concerned with the diffusion of oxygen in zirconium over the temperature range 400 to 585°C and its relation to oxidation and corrosion. Scaling behavior of zirconium in air and oxygen was investigated by Case, whereas BMI studied zirconium-water reactions at subatmospheric pressures. (W. K. Boyd)

Aluminum Corrosion

Considerable effort has been directed toward improving the corrosion resistance of aluminum and its alloys to high-temperature water and steam. Aluminum has adequate resistance at temperatures below 400°F; above this temperature, however, the metal suffers rapid intergranular disintegration. To prevent this and improve general corrosion, several techniques have been employed, namely, alloying with nickel and iron and the use of corrosion-inhibitor treatments, with the latter receiving increased attention.

Knolls[6] reports that as little as 0.1 wt. % sodium silicate will inhibit corrosion of 1100 aluminum (commercially pure grade) in water at temperatures up to 400°F. At 480°F, corrosion rates increase exponentially with time, both with and without silicate additions. Data show, however, that initial corrosion rates in solutions containing 0.1 to 1.0 wt. % silicate are of the order of $^1/_{100}$ of that in pure water. After 350 hr, the rate in the silicate solution approaches that obtained in the first 24 hr of exposure in pure water.

It was also found[7] that service life of aluminum and its alloys in high-temperature water could be extended by additions of phosphoric acid (pH = 3.5) or polyhydroxyanthraquinone (alizarin). A 500-ppm addition of the organic reduced the corrosion at 680°F to practically zero. Life of the alizarin was found to be good, no significant thermal decomposition being detected after one week of exposure. Radiation stability of the organic, although not known, is not expected to be good. Small additions of $MgHPO_4$ also inhibit corrosion of aluminum in high-purity water. Results of studies at ANL[8] indicate that corrosion rates of the nickel-bearing M-388 alloy were of the order of 1.3 mg/(dm^2)(day) in 550°F water containing 5 ppm PO_4 as $MgHPO_4$.

Another problem that is constantly receiving attention is that of methods of measuring corrosion of aluminum. A recent Hanford publication[9] discusses several techniques for removing corrosion products from aluminum and measurement of corrosion rates without stripping the oxide film. A device called the "Corrometron" is described. This instrument consists of two 1-in. micrometers arranged in such a manner as to permit measurement of the thickness of the base metal after corrosion testing. Its operation depends upon electric conductivity of the base metal. Although the instrument has been successfully employed, it appears that considerable skill must be acquired to ensure satisfactory operation.

Also of interest to the reactor-corrosion engineer, is the Corrosion and Wear Handbook for Water-cooled Reactors.[10] This book presents background information and general principles for the benefit of those who are encountering, for the first time, some of the problems associated with the use of high-purity water as a reactor coolant. (W. K. Boyd)

Niobium, Molybdenum, Chromium, and Nickel Corrosion

Recent research on gas-metal reactions has been concerned with niobium and niobium alloys. Pure niobium oxidizes linearly at rather rapid rates above 500°C, although additions of molybdenum, vanadium, and titanium allow formation of protective scales at 1000°C. Recent work at BMI[11] indicates that these alloys oxidize much less rapidly in air at 1000°C than pure niobium,

Table V-1 OXIDATION RATES FOR Nb AND Nb ALLOYS

Composition, wt. %	Parabolic rate, $mg^2/(cm^4)(hr)$	Weight gain in 10 hr, mg/cm^2
Nb*		301
Nb–7.7 Mo	41.2	20.3
Nb–7.3 V	42.9	20.7
Nb–14.7 Ti	51.2	22.7

* Linear rate for niobium is 30.1 $mg/(cm^2)(hr)$.

as seen in Table V-1, but the rates are still quite high compared to commercial heat-resistant alloys of the iron- or nickel-base type.

No solution to the problem of catastrophic oxidation of molybdenum by alloying has been published, but work at BMI over the past several years has shown that defects in nickel-base claddings on molybdenum are self-healing through the formation of nickel molybdate.[12] It was revealed recently that General Electric Company has successfully operated a jet engine using molybdenum turbine buckets.[13] This, of course, implied the use of a successful oxidation-resistant coating or cladding on the molybdenum. Molybdenum and niobium have been suggested as fuel-element materials for a high-temperature helium-cooled reactor.[14]

Results of recent studies of the oxidation behavior of pure nickel and chromium by Westinghouse Research[15,16] indicate that a protective film is formed on nickel up to 1000°C, but cracking of the film destroys its protectiveness at higher temperatures. Cracking can be reduced by proper alloying. Chromium forms protective films up to 900°C, but, above this temperature, vaporization of chromium metal destroys the film and increases the oxidation rate. This behavior may prevent the development of chromium-base oxidation-resistant alloys above 900°C.

The NBS has determined the oxidation rates[17–19] of nickel–20 wt. % chromium, iron–25 wt. % chromium–5 wt. % aluminum, and

Table V-2 AIR-OXIDATION RATES FOR NICKEL- AND CHROMIUM-CONTAINING ALLOYS*

Composition, wt. %	Test temp., °C	Parabolic rate, $mg^2/(cm^4)(hr)$	Weight gain in 10 hr, mg/cm^2
Ni–20 Cr	982	0.00365	0.191
	1093	0.0297	0.546
	1204	0.13	1.14
Fe–25 Cr–5 Al	982	0.00166	0.129
	1093	0.0089	0.298
	1204	0.039	0.625
Ni–15 Cr–3 Al–0.5 Ti (Inconel 702)	871	0.00097	0.099
	982	0.0064	0.254

* Data from National Bureau of Standards, Washington, D. C.[17–19]

Inconel 702. These alloys all have good oxidation resistance in the 1000 to 1200°C range, the best being iron–25 wt. % chromium–5 wt. % aluminum, as seen in Table V-2. However, they have lower strengths at high temperatures than niobium and molybdenum and will be most useful

as claddings for higher strength alloys which do not have adequate oxidation resistance.

(W. D. Klopp)

Although nickel has been found to suffer less corrosive attack by fused sodium hydroxide (an interesting moderator-coolant) than any other material tested to date, mass transfer in high-temperature, dynamic, polythermal systems has prohibited its use. In a recent National Advisory Committee for Aeronautics (NACA) report,[20] several ideas are presented about the mechanism of thermal-gradient mass transfer in the NaOH-nickel system. Mechanisms discussed, including physical, electrochemical, and chemical, are evaluated in the light of theoretical and experimental evidence.

The mechanism given as being the most probable involves the formation of Na_2O by dissociation of NaOH (admittedly slow), subsequent reaction of the Na_2O with nickel in the hot zone to form sodium nickelite (Na_2NiO_2) and free sodium, and the reversal of this reaction in the cold zone to precipitate free nickel. (J. H. Stang)

Corrosion Mechanisms

Mechanism of Corrosion of Fuel Alloys

One of the limiting factors in the use of solid uranium and thorium fuels in power reactors is their poor corrosion resistance in oxidizing mediums. Elaborate precautions are taken to provide a protective envelope around the fuels, not only to prevent fission products from escaping into the coolant stream but also to protect them from corrosive attack. In the pressurized water reactor field, relatively corrosion-resistant alloys have been developed as further protection in the event of a leak in the protective envelope. The alloys possess certain limitations, and those of high uranium content — the gamma phase alloys — suffer a cracking or oxidation type of failure upon continued exposure in high-temperature water.

An understanding of the mechanism of the corrosion reaction might indicate what steps should be taken to improve corrosion resistance. A considerable amount of this type of work is being conducted on uranium-water and uranium-oxygen reactions.

The generally accepted reaction for uranium with degassed water has been the formation of uranium dioxide and the evolution of hydrogen. However, the amount of hydrogen evolved has never equaled the stoichiometric quantity that should have been formed if UO_2 were the corrosion product. The theory has been advanced that an intermediate reaction occurs in which the liberated hydrogen combines with uranium to form a hydride. The hydride then reacts with water and is oxidized to UO_2. Battelle[11,21-23] has found no evidence of uranium hydride in the corrosion product formed in water and steam at temperatures up to 200°C. Upon removing the corrosion product from the metal as it is formed in 100°C water and quenching it in a colder zone, an average valence of 3.16 was found for the uranium in the corrosion product. It is believed that the low valence indicates the presence of hydroxides of uranium having +2 and possibly +3 valence. The hydroxides of uranium having lower valence then progressively oxidize to the +4 state, with UO_2 being the end product. It was postulated that hydroxides are present in the corrosion product along with UO_2 and that a material balance between hydrogen evolved and metal oxidized must consider the hydroxides.

Workers at ANL[24] have recently reported on the effect of hydrogen in uranium on the corrosion behavior of uranium in boiling water. They had previously demonstrated that the corrosion resistance of plate-grade uranium quenched from the beta or gamma phase approached that of high-purity uranium. However, outgassing observed at the alpha-beta transformation temperature during heat-treatment suggested that a hydride might be trapped in the alpha lattice. The hydride could be responsible for the higher corrosion rates of alpha-heat-treated uranium. However, the introduction of hydrogen into gamma-heat-treated uranium failed to increase its corrosion rate. They concluded that the lower corrosion rates obtained with gamma-heat-treated uranium still could be best explained on the basis of solution of metallic impurities. The latter were believed responsible for the poorer corrosion resistance of alpha uranium.

In addition to water-uranium reactions, there have been recent reports on the oxidation of uranium. Shaw and Hindmarch,[25] at Windscale, England, have studied the oxidation behavior of uranium over the temperature range of 300 to 900°C in argon containing up to 50 ppm oxygen. They attribute low oxidation rates at 300°C and low oxygen contents to the rapid formation of a protective film, with subsequent oxidation by

diffusion. At the higher temperatures and higher oxygen levels, the protective film reaches a certain thickness at which it fissures, exposing the underlying metal to oxygen. The oxidation rate then becomes linear with time.

Westinghouse Research[26] has studied the oxidation of the 50 wt. % uranium–zirconium alloy that has been considered as a fuel alloy for pressurized water reactors. Uranium enrichment of the oxide was found, which indicated migration of uranium into the oxide layers. Large voids were found in the oxide film oriented parallel to the oxide-metal interface. The voids apparently admitted oxygen, thus accounting for the linear oxidation rates obtained. The voids were believed to be a result of periodic blister formation. Activation energies of 19 to 23 kcal/mole were obtained for oxidation in the range of 300 to 500°C. (W. E. Berry)

Metal-Water Reactions

A series of BMI experiments were reported[27] on the reactions of Zircaloy-2 and Zircaloy-B with water and steam. In addition to reaction rates radiation emissivities and diffusion rates of oxygen in Zircaloy were also measured. The data for the reaction of solid Zircaloy-2 with steam at 50 psia, over a temperature range of 1000 to 1690°C, were best correlated on the basis of a second-order reaction

$$v^2 = 0.1132 \times 10^6 e^{(-34{,}000 \pm 1440)/RT}\, t$$

with v in milliliters of hydrogen per square centimeter and t in seconds. The reaction of single, molten Zircaloy-2 droplets of about 0.2 in. diameter with hot water showed that 39 to 45 wt. % of the Zircaloy-2 could react.

Later experiments on uranium and on uranium-niobium and uranium-niobium-zirconium alloys are reported.[28] Over the temperature range of 800 to 1690°C, it was found that solid uranium was the most reactive (with steam at 50 psia) of all materials studied, with the exception of the solid 75 wt. % (uranium–10 wt. % niobium)–25 wt. % Zircaloy-2 alloy, which behaved in an anomalous fashion. The solid uranium–10 wt. % niobium alloy had the lowest reactivity, and the solid 50 and 25 wt. % (uranium–10 wt. % niobium) alloys had reaction rates with steam intermediate between uranium and the uranium–10 wt. % niobium alloy and comparable with those[27] of solid Zircaloy-2.

As reported by Aerojet Projects, Inc.,[29] the molten metals, zirconium, Zircaloy-2, aluminum, uranium, sodium-potassium alloy, and stainless steel, were sprayed into water. It was found that the reactivity of molten aluminum is nil at temperatures up to 1170°C. At temperatures above 1170°C, the reaction becomes increasingly more complete. At a temperature of 2200°C, aluminum was found to be a more powerful reactant than zirconium. Stainless steel was slightly less powerful than zirconium under these conditions. In a study of the oxidation rates of Zircaloy-2 at temperatures up to 1200°C in high-pressure steam (up to 1000 psi), it was determined that pressure has little or no effect on the corrosion rate of Zircaloy-2.

Recent work by Ames[30] on the reaction between magnesium and water vapor and that of BMI[31] on the reaction between zirconium and water vapor at subatmospheric pressures will prove helpful to future investigators in this field.

Two types of empirical experiments have been performed. Studies were made by Aerojet on the effects of dropping a quantity of molten metal into a pool of water. In some cases a blasting cap was detonated to aid in dispersing the molten metal. Although some results of these tests have been reported earlier, additional data are given.[29] On the basis of measurements of oxide-film thicknesses from photomicrographs, it was calculated that 25 per cent of a molten metal sample of Zircaloy-B with 0.9 wt. % beryllium had reacted. Although these tests were not conclusive because less than one-third the original sample was recovered in any one test, pure zirconium appeared more reactive than the Zircaloy B–0.9 wt. % beryllium alloy.

Tests reported by BMI[27] were made in an attempt to simulate the phenomena that would occur during the melting of a fuel-element plate assembly in a steam atmosphere. Although the tests were only partially successful because of the large heat losses from the electrical-resistance-heated assembly, it was concluded that blobs rather than small droplets of molten metal would be produced in this fashion, thus minimizing the amount of reaction that would occur in the molten state.

Alcoa[32] recently reported tests made by dropping molten aluminum into water. For molten-metal temperatures from 670 to 900°C, it was concluded that the exothermic chemical reaction of aluminum with water played a minor part, if any, in the resulting explosions. There was no

evidence whatever of a hydrogen explosion or of a mechanical restraint of expanding steam by solidifying metal. (A. W. Lemmon, Jr.)

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Radiation Effects in Fuel Materials

Of primary concern in the study of radiation effects on fuel materials are the changes produced in the materials by fission products. These effects can be divided into two general categories: (1) the changes produced by collision of the energetic fission fragments with the constituent atoms of the fuel and (2) the changes resulting from the gradual accumulation of quantities of fission products and their diffusion in the parent fuel material. Both effects are reviewed by Gurinsky and Dienes,[1] and the effect of collision of energetic heavy particles is discussed in detail by a number of authors.[2–4]

Much of the experimental work on fuel materials deals with changes in physical and mechanical properties resulting from these effects of radiation. This is discussed in other sections dealing with the appropriate materials. This section deals only with a review of that information for which the authors have sought to interpret the over-all changes produced in terms of solid-state theory or have conducted experiments specifically designed to clarify the mechanism by which radiation-induced changes occur.

The growth of uranium specimens during irradiation has been known for some time and has been the subject of a considerable amount of experimental work. A number of mechanisms have been proposed by various investigators to account for this behavior.[1] According to one theory the growth of uranium is due to migration of lattice vacancies and interstitial atoms generated by fission fragments. According to this theory the vacancies and interstitial atoms may be eliminated by migration to grain boundaries and free surfaces. Therefore growth should be proportional to grain size. The observed growth of alpha uranium in preferred crystallographic directions is attributed to anisotropic diffusion.

A program is in progress at Sylvania to test the above theory.[5,6] This program consists of two phases: (1) study of the effect of grain size on growth of alpha uranium during irradiation and (2) measurement of the anisotropy of self-diffusion in alpha uranium.

To determine the effect of grain size on growth, specimens of alpha uranium having identical preferred orientation but different grain sizes were irradiated. It was found that grain size has a smaller effect than anticipated. The authors conclude that interstitials and vacancies are only partially eliminated at the grain surfaces and suggest that the following mechanisms may also be operative: (1) absorption of interstitials and vacancies on lattice dislocations, (2) precipitation of vacancies as macroscopic voids, and (3) precipitation of interstitial atoms as extra planes of atoms.

The second phase of the study on anisotropy of self-diffusion is apparently still in progress. It was planned to study self-diffusion in alpha uranium by placing a film of U^{233} on a single crystal of alpha uranium. The alpha activity of U^{233} is to provide the means for following the extent of diffusion. The experimental techniques involved are quite complex. It appears from the latest report located,[7] that all the experimental difficulties have not yet been resolved.

As part of the program the diffusion of gold in alpha uranium was studied to serve as a check on the experimental techniques. A diffusion coefficient of 1.4×10^{-14} cm^2/sec was determined at 650°C. Based on the generalization that diffusion coefficients of dilute elements are expected to be similar to those for self-diffusion, it appears that self-diffusion of uranium may be about an order of magnitude lower than anticipated.

The release of fission gases from reactor fuels can prove to be a serious problem, particularly at high temperatures, and has been the subject of some study, especially in the case of UO_2. Two mechanisms exist by which fission gas may be released from a fuel, recoil and diffusion. For UO_2, Bettis[8] concluded that fission-gas release is primarily by solid-state diffusion.

Two techniques have been utilized to study fission-gas diffusion. One technique consists in dissolving a noble gas or gases in a fuel material and measuring the rate of release; the other involves measuring the release of fission

gases during irradiation and during postirradiation heat-treatment.

In a study at Bettis[9] the diffusion of helium that had been dissolved in 0.15 μ UO_2 powder was measured. The diffusion coefficient at 1100°C was found to be 1.2×10^{-15} cm^2/sec. Attempts were made to conduct similar experiments using neon, argon, krypton, and xenon. However, no measurable quantities of the latter three gases could be dissolved at the experimental conditions.

In another study[8,10] involving measurement of release of fission gases from irradiated UO_2, it was found that the activation energy for diffusion of fission gases is very similar to that for diffusion of oxygen in UO_2 and is less than one-fourth that for diffusion of uranium in UO_2. This suggests that diffusion of fission gases in UO_2 may be related to the oxygen-ion diffusion mechanisms. In addition, it was found that the diffusion coefficient for fission gases is about six orders of magnitude lower than that for oxygen-ion diffusion. This was attributed to the difference in atomic size, 1.90 A for xenon and 1.69 A for krypton, compared to 1.32 A for the doubly-charged oxygen ion.

Although there is a considerable scatter in the data obtained on fission-gas release, the results are consistent with a diffusion mechanism. If this is the correct mechanism, it is clear that such factors as density of UO_2 and time and temperature of irradiation are important in determining the quantities of gas released.

Several interesting comparisons between solid-state theory and experimental observations on structural changes of fuel materials appear in the recent literature. Good correlation was obtained between the observed degree of homogenization of uranium-molybdenum alloy during irradiation and a theoretical treatment based on the thermal-spike concept.[4,11]

Investigators at Bettis[10] have been successful in explaining reversion of alpha-transformed uranium-molybdenum and uranium-niobium alloys to the gamma-phase alloys during irradiation on the basis of the displacement-spike mechanism.[12]

Also, the saturation of density decrease and hardness changes at low burn-ups in the case of epsilonized U_3Si appears to be explained satisfactorily on the basis of complete disordering of the U_3Si which was predicted to occur at a very low burn-up.[10]

The increasing efforts to correlate experimental observations with solid-state theory and the degree of success in obtaining a correlation, at least in some cases, are encouraging. It is to be hoped, that, through such efforts, it may be possible at some future time to predict the behavior of certain fuel materials.

(P. Schall, Jr.)

Radiation Effects in Nonfuel Materials

Basic Studies

Recent additions to a basic knowledge of the effects of irradiation by charged particles and neutrons range from irreversible changes due to thermal spikes to the motion of defects at low temperature. Several correlations of different methods of measuring defect concentration have been reported. The results emphasize the need to provide known particle and/or neutron energy and to simultaneously control the rate of annealing, as a basis for comparison with theory.

The temperature distribution around a primary knock-on as a function of radius and time has been calculated by Oak Ridge.[13] The estimates of time at temperature in a thermal spike are related to the time required for diffusion and nucleation of irreversible changes. Accordingly, it is the slower rate of temperature change in the tail of a temperature spike that generally is significant. The calculations yield fair agreement with reported data on precipitation of the nickel-beryllium compound from nickel solid solution and on the instability of austenitic type 347 stainless steel. In work on etching of irradiated germanium, it has been suggested that a heterogeneity of lattice disorder, as indicated by etch delineation of a third-order network (presumably subgrain structure), was produced by thermal spikes.[14] However, it may be preferable to consider the cause as the displacement spike region at the end of a thermal spike. The agreement of the activation energy (about 1.7 ev) for annealing of the etching effects with the results of prior work on electric conductivity indicates that both methods are observing the same process.

The continuing studies of low-temperature irradiation of metals are of benefit in leading to a fundamental understanding of the nature of defects and their mobility. Studies at the Uni-

versity of Illinois[15] have shown good correlation of the lattice expansion for deuteron irradiation near 10°K with electric-conductivity change. However, a factor of 10 difference in number of defects calculated from theory and determined from lattice expansion was found. Data on annealing of the lattice expansion in copper are in general agreement with the results of workers at Oak Ridge on electric conductivity.[16] There is 40 to 55 per cent recovery from about 20 to 50°K, followed by slow recovery up to 300°K. The lattice-expansion effects are primarily a result of the number of interstitials produced, and results[15] indicate greater recovery as determined by lattice contraction up to 50°K than by electric conductivity. This could indicate loss of interstitials at a rate different from vacancies and therefore not a simple interstitial-vacancy recombination. The conclusions of Oak Ridge workers that a crowdion is the mobile defect at these low temperatures are supported by Illinois data. The crowdion is a line defect produced by an interstitial distorting several atoms in a row, with a low activation energy for diffusion in a line. Thus the concept is set forth that, for low-temperature irradiation and annealing, the crowdion can move distances up to 1000 atomic diameters. The stopping of a crowdion in a dislocation or other boundary could then produce the elimination of interstitials without concomitant loss of vacancies.

Recent work at Hanford[17] gives data supporting the lower energy for diffusion of interstitials as compared to vacancies. Their data for molybdenum and zirconium show a continual increase in strain as measured by X-ray diffraction broadening but with the lattice expansion passing through a maximum. This would indicate an increasing ratio of vacancies to interstitials.

(A. E. Austin)

Empirical Studies

Several reports have been made on the effects of irradiation on the strength properties of various types of metallic materials. In general, these reports have confirmed what has been reported previously, i.e., that yield strength and tensile strength are increased and that elongation and reduction of area are decreased by neutron irradiation. Some materials not previously studied are being irradiated and tested for changes in strength properties by Phillips Petroleum Co.[18] Hastelloy X, type 410 stainless steel, 16-1 Croloy, 2¼ Croloy, Inconel 702, and K monel are under test in the Materials Testing Reactor. Because of corrosion difficulties stemming from failure of the nickel plate, irradiation of 2¼ Croloy was discontinued after four operating cycles. Integrated fluxes on the materials tested are not yet available, but, after exposures up to 84 days, the Hastelloy X, Inconel X, and type 410 stainless steel showed considerable increases in yield strength. The yield strength of Hastelloy X was doubled, with only insignificant reduction in elongation and an actual increase in reduction of area. In the case of Inconel X and type 410 stainless steel, there was a significant decrease in elongation and little or no change in reduction of area.

Information reported on Zircaloy-2 shows that, after irradiation to an integrated neutron flux of 1.4×10^{20} (1 mev average neutron energy), yield strength is doubled,[17,18] and a yield point is developed.[19] This yield point can be annealed out by a postirradiation heat-treatment of 300°C. In 0.040-in.-diameter zirconium wire that was heavily cold-worked prior to irradiation, the original room-temperature yield strength, 107,000 psi, was unaffected or slightly decreased by irradiation[20] to 5×10^{19} nvt.

Reports[21] of tests on irradiated samples of construction steels show that irradiation to 3.8×10^{18} nvt at 500 to 700°F had little effect on the Charpy V notch transition temperature of ASTM A302B steel. This radiation level is apparently too low to produce gross effects in this manganese-molybdenum steel, or the temperature at which irradiation was carried out was high enough to anneal out gross effects. Bend tests on samples of ASTM A201 steel irradiated at about 400°F to integrated fluxes as high as 4.3×10^{20} indicated that rather severe embrittlement of this steel could be considerably reduced by a postirradiation anneal at 600°F for 1 hr.

A report[22] that precipitates in iron-copper alloys are redissolved under irradiation indicates that perhaps the properties of precipitation-hardened materials might be changed drastically by irradiation. Their observation of a homogenizing effect of radiation correlates with observations at Bettis that homogenization occurs in partially transformed gamma-phase alloys, leading to stabilization of the gamma phase.

(D. C. Martin)

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